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Summary of Characterization Tests  
on a Low Density Epoxy Grout for Use as a  
Bulkhead Seal Material in the  
US Strategic Petroleum Reserve

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ABSTRACT

Flow and mechanical characterization tests are used to evaluate a low density epoxy grout for potential use as a bulkhead seal material in the SPR Weeks Island Service Shaft. The grout is required to float on crude oil, spread around numerous oil withdrawal pipes in the shaft, completely cover the crude oil surface, and cure to form a structurally stable plug unto which non-buoyant grout material may be added. A series of completed bench scale flow tests and a quarter scale simulation of the Service Shaft are described. The interfacial shear strength of the low density epoxy grout on steel and salt was tested at various cure times and interface conditions--dry and oily. The tensile strength and modulus of the epoxy grout was tested at various cure times. Test results are compared to material requirements to qualify the low density epoxy grout for use in the underground shaft and potentially the two raisebores at Weeks Island. Quality control tests and a permeability verification test are developed as part of a construction specification for the grout.

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Pete Rand, with assistance from Orelia Montoya (both of 1813), performed the bench scale flow tests (Section 3.1) and evaluated the results of the quarter scale test. Pete contributed expertise in polymers and advice throughout this project and developed quality control tests for later use. Orelia prepared the epoxy specimens used in all the bench scale tests and performed many tests.

Jeff Lowry of Wil-Cor, Inc., formulated the epoxy resin system used in all the tests and was responsible for the quarter scale pretests (Section 3.2) and quarter scale test (Section 3.3). He and his coworkers are credited with fabricating and performing the quarter scale test in Pasadena. Texas.

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Wayne Gravning (3153) videotaped and photographed the bench scale flow tests at Sandia and the quarter scale test and pretests in Texas. Cathy Ehgartner (6321) edited and helped in preparing this report for publication.

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## 1.0 INTRODUCTION

The Strategic Petroleum Reserve was created to reduce the vulnerability of the United States to interruptions by foreign oil suppliers. Approximately 670 million barrels of crude oil are presently stored underground in salt domes at six sites located along the Gulf of Mexico. Most of the crude oil is stored in leached caverns, but at Weeks Island, Louisiana, an underground mine was converted for use.

Concrete bulkheads installed in the underground mine accesses, which include shafts and raisebores, isolate the oil-filled underground workings from the overlying facility drifts. Figure 1-1 shows the underground facility and location of the bulkhead seals. Underneath, two levels of room and pillar workings at elevations of 500 and 700 ft store approximately 72 million barrels of crude oil. Recent bulkhead inspections showed some cracking of the concrete. As a result, structural design analyses were performed (Blanford, et al., 1990). The analyses conservatively assumed loading under a mine flooding scenario. As a result of the inspections and updated design analyses, the bulkhead seals in Raisebores No. 1 and 2 and the Service Shaft require remedial work. Figure 1-2 shows the bulkhead seal in the Service Shaft. The bulkhead seals in the 6 ft diameter raisebores are similar, but lack the oil withdrawal pipes.

Because of the inherent risk and high costs associated with conventional mining and concrete placement in the shafts and raisebores, the use of a low density epoxy grout was investigated. The grout material would be pumped down (60 - 70 ft) through the existing bulkheads to the crude oil level, where it would float and harden to form a plug onto which more grout could be added. Additional grouts and cements would then fill the shaft up to the existing bulkhead level, resulting in a much more massive bulkhead seal than currently exists. In comparison to conventional mining techniques, this procedure for upgrading the shaft's seal capability is considerably less expensive, reduces the vulnerability of a crude oil spill during the upgrade, and worker safety is improved. Conventional practices would require a mined opening to

access the underside of each bulkhead, the construction of supports and forms, and placement of concrete. Because the bulkhead seals are temporarily bypassed, they would be useless should flooding occur during the upgrade. Also, work crews would be operating over several hundred feet of crude oil.

The initial layer(s) of epoxy grout must be capable of floating on crude oil and spreading around multiple pipes within the Service Shaft to form a structurally stable plug. This epoxy is known as the "low density" formulation, and the material requirements are discussed in Chapter 2. Additional epoxy grout and cement used in the overlying lifts will be denser, non-buoyant materials with improved sealing characteristics and less costly to manufacture. These materials are referred to as "high density" in this report.

The low density epoxy grout must be carefully formulated and well characterized because oil **drawdown** could be jeopardized if it were to sink or if the plug it forms were to leak the high density grout. The primary oil withdrawal sump is located directly beneath the work area at the bottom of the shaft. The ability of the plug material to float on crude and spread among the oil withdrawal pipes of the Service Shaft are tested by the flow experiments described in Chapter 3.

The grout plug must structurally support the weight of the high density grout lift, and the grout is expected to bond to the steel pipes and salt wall rock of the shaft. Bonding to the salt and steel will not only stabilize the plug structure, but will also eliminate interfacial flow between the overlying heavy grout and crude oil. The mechanical strength of the grout and its bond to salt and steel are quantified by the mechanical strength tests described in Chapter 4.

Chapter 5 concludes the report and discusses quality control and verification tests to assure that the low density epoxy plug will perform in the mine as intended. A construction specification is developed in the Appendix which details procedures for qualifying a grout for use, handling and quality control tests, pouring the plug, and testing the plug for leakage.



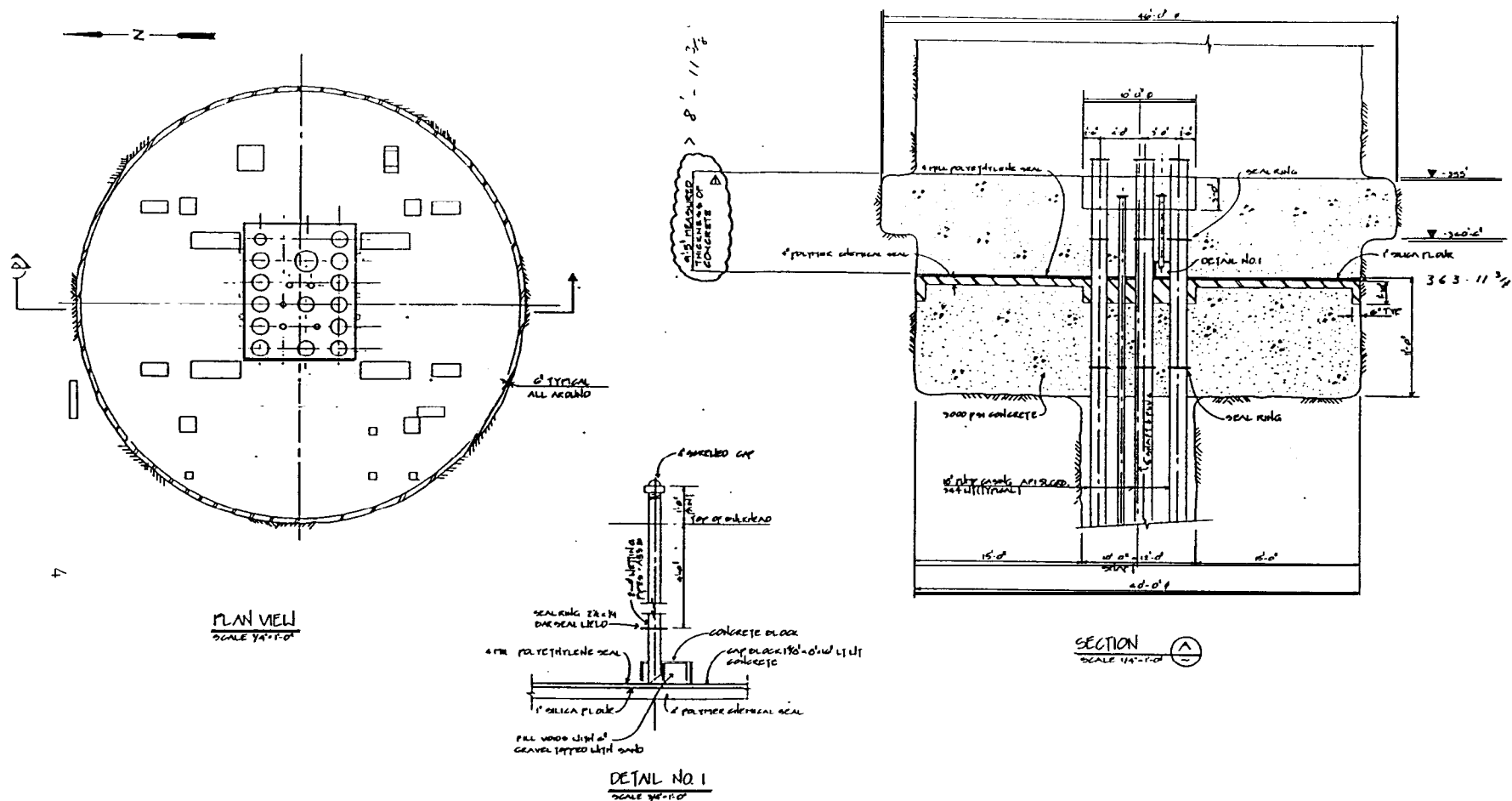


Figure 1-2. As-Built Drawing of Service Shaft Bulkhead.

## 2.0 MATERIAL REQUIREMENTS

The primary function of the low density epoxy grout plug is to retain the high density grout pour until it hardens. To accomplish this, the grout material and the plug that it forms must exhibit certain characteristics. In this chapter, general requirements, and later more specific requirements, are defined.

The low density epoxy grout material and the plug that it forms must meet the following general requirements.

1. The initially placed grout must float on the crude oil which has a density of approximately 55 lb/ft<sup>3</sup>.
2. The grout must spread across the oil surface to cover the entire surface area of oil in the shaft or raisebore. The 6 ft dia raisebores are smaller than the Service Shaft and do not contain any piping. Therefore spreading is more easily accomplished in the raisebores than in the Service Shaft. The Service Shaft is 10 ft by 12 ft rectangular with 21 steel pipes descending through the existing bulkhead to a sump at the bottom of the mine from where the oil is withdrawn. The numbers and diameters of piping are: one 24 in, thirteen 18 in, one 12 in, and one 6 in. These comprise approximately 24 percent of the cross-sectional area of the shaft (see Figure 1-2).
3. The grout plug must contain the high density grout pour. The plug must have a low permeability to the high density grout and be able to structurally support it. In order to contain the high density grout pour, the plug should bond to the steel pipes and salt. No significant cracks should develop either along interfaces or within the grout after curing. During curing the grout will generate heat and later it will cool to ambient temperature. As a result of thermal expansion and contraction, cracks may develop. The oil level is not expected to fluctuate during curing of the plug, hence the plug must sustain only the weight of the immediate overlying

grout lift as buoyancy forces will balance the weight of the low density plug. The grout plug will tend to bend, and the bond interfaces with salt and pipes will tend to shear under loading.

4. Materials added to the shaft must not interfere with the oil withdrawal capability of the facility. Chemical interactions, dissolution, or segregation of the grout or its components must not result in pieces of material that could sink to the sump and interfere with drawdown. Chunks of floating material are also prohibited as they may interfere with **drawdown** in its final stages when the oil surface is near sump level. The grout must not corrode the steel withdrawal pipes of the Service Shaft.
5. Grout must be available at reasonable costs and the placement capability (mixing and pumping) commercially and readily available. No unsafe or hazardous conditions or events should arise from the use or placement of the grout material.

In consideration of the above general requirements, more detailed requirements evolved. These are listed in Table 2-1 and later in the Contract Specification provided as an Appendix to this report.

The strength requirements of the grout and its bond to salt and steel were derived from three engineering calculations. The first calculation assumed that the only bonding to occur will be with the wall of the Service Shaft. Knowing the circumference of the shaft wall (44 ft) and the estimated weight of the overlying epoxy ( $95 \text{ lb/ft}^3$ ), and lift size (1 ft), the required shear strength of the bond to salt was calculated at approximately 2 psi. The second calculation assumed bonding with the pipes only with a total pipe perimeter of 79 ft. The required shear strength of the bond to steel was calculated as approximately 1 psi. If bonding were to both the steel pipes and salt wall, the required shear strength of the bond would be much less than 1 psi.

**Table 2-1**  
**Grout Material Requirements**

- 
1. Grout can be mixed, pumped, and delivered down to the oil surface without altering the characteristics which qualified it for use.
  2. Grout shall have a specific gravity that is less than 45 lb/ft<sup>3</sup>.
  3. Grout shall not be affected by the crude oil. Resin, filler or aggregate, or any other component of the grout shall not separate from the grout and sink into crude oil. Crude oil shall not destabilize the mix and cause any material to sink into the crude oil.
  4. Grout shall have a fluidity that allows it to spread across the surface of the crude oil from the injection point(s) and flow around all vertical pipes in the Service Shaft, self-level, and completely cover the entire oil surface.
  5. Grout shall harden within 24 hrs of the pour and be ready to accept the next grout (low or high density) pour.
  6. Grout shall develop a minimum shear bond strength to salt and steel of 10 psi 24 hrs after mixing for both clean dry and oil coated interface conditions.
  7. Grout shall develop a minimum tensile strength of 10 psi 24 hrs after mixing.
  8. Grout shall not shrink more than 1.0 percent in a 24 hr period or develop any cracks.
  9. Grout shall be formulated such that "runaway" or uncontrolled reactions do not occur.
  10. Grout shall not cause temperature of crude oil to exceed 150°F due to heat released from the epoxy.
  11. Grout parts or packages shall have a shelf life of at least two weeks from date of manufacture.

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In addition to the above material requirements, the grout plug itself must pass a permeability test as described in Attachment 4.02 to the Appendix.

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The third engineering calculation examined bending stresses of the epoxy grout plug. The plug was assumed to freely deform across the 12 ft shaft span, when in reality the span will be much less due to the piping. The same 1 ft lift load ( $95 \text{ lb/ft}^3$ ) was applied to the plug to determine a minimum required tensile strength of 5 psi for the grout. No credit was taken for any buoyancy forces that will act on the underside of the plug.

The above engineering calculations, although conservative in the assumptions made, do not incorporate a safety factor. Therefore, for purposes of this report, the required strength of the low density epoxy grout and its bond to either salt or steel is 10 psi. A safety factor is also incorporated into the density requirement ( $< 45 \text{ lb/ft}^3$ ). Because of potential uncertainties and variations in grout density, the maximum allowable density is  $10 \text{ lb/ft}^3$  less than the expected density of the crude oil ( $55 \text{ lb/ft}^3$ ).

A low density epoxy grout potentially capable of meeting all of the above requirements was formulated by Wil-Cor, Inc., of Pasadena, Texas, and procured for testing. The material is a two part proprietary formulation named "Armor Plate Ultra Low Density Epoxy Grout 1000." The same basic formulation of low density epoxy grout was used in all the experiments; however, the relative percentages of ingredients were varied over the time that these experiments took place. The flow tests are discussed in Chapter 3 and the mechanical tests in Chapter 4.

### **3.0 FLOW CHARACTERIZATION EXPERIMENTS**

Lab or bench scale experiments are discussed in Section 3.1 followed by the quarter scale pretests in Section 3.2, and the quarter scale simulation of the Service Shaft in Section 3.3. Within each section, the purpose, procedures, and results of the test are discussed.

#### **3.1 Bench Scale Tests**

##### **3.1.1 Purpose of Bench Scale Tests**

Two series of lab scale experiments were performed. The first series used a small batch of grout to test for floatation and chemical interaction with oil and water at different temperatures. These experiments were the first assessment of the material characteristics and hence are considered as preliminary.

The second series was more comprehensive and the scale slightly larger (approx. 6 in). The ability to float, self-level, and not interact with crude oil were examined along with the ability to cover an area containing pipes.

##### **3.1.2 Bench Scale Test Procedures**

The first series or preliminary experiments were performed at both room temperature and at elevated temperature (85°F). The epoxy was floated on water, vacuum oil, and crude oil and visually examined for evidence of dissolution or chemical interaction one day and one week after the start of the experiments. The epoxy was introduced by hand into 3.5 in petridishes.

The second series of tests and their purposes are described in Table 3-1. The tests were performed at the expected mine temperature (80°F) in 6 in dia beakers. The steel piping in Test 3 consisted of 15 3/4-in and 5 1/4-in dia pipes. The epoxy was mixed by hand as shown in Figure 3-1 and then poured into a Cemco (Figure 3-2) which injected the epoxy

onto the crude oil through a 1/4 in dia outlet at constant pressure. Figure 3-3 shows a typical experimental setup with the Cemco positioned over the crude oil filled beaker on a scale. The Cemco was used to inject the grout into the crude oil.

Table 3-1  
Description of Bench Scale Flow Tests

Test	Name	Test Procedure
1	Float/Precipitates	Float epoxy on crude over water and observe precipitates, if any, in water. Inspect sample for dissolution. Sample should be buoyant not touching edge of beaker if possible.
2	Leveling	Slowly introduce epoxy onto crude at edge of beaker and observe ability to spread and self-level.
3	Viscosity	Same as Test 2 except beaker contains piping scaled down and spaced to represent layout in Service Shaft.

### 3.1.3 Bench Scale Test Results

Results from the preliminary first series experiments were encouraging. The epoxy floated and did not appear to interact with the crude. The density of the epoxy was measured at 44.2 lb/ft<sup>3</sup>. Spreading of the samples on crude resulted in the thickness of the specimen at approximately one-fifth the diameter. Temperature differences did not result in any notable differences. Electron microscope scans of a fractured surface of the grout at low and high magnification are shown in Figures 3-4 and 3-5. The epoxy resin and distribution of particle sizes are evident in the scans. Particle sizes ranged from approximately 0.05 to 0.3 mm.

The results for Tests 1 through 3 of the second series were videotaped on February 18, 1991 (Gravning, 1991a). The early time results (5 min

after pour) of the three tests are shown in Figures 3-6 through 3-10. The epoxy in the floatation test floated and spread with no apparent reaction, either chemical or mechanical, with the crude. The epoxy for the leveling test spread and leveled to a constant thickness of approximately one-half inch. Approximately the same amount of epoxy used in the leveling test was used in the viscosity test. The epoxy spread and filled around the pipes to eventually cover all surface area. The epoxy layer in the viscosity test leveled across the top, but the layer thins in confined areas such as between the glass and the pipes. The minimum thickness was approximately 50 percent of the overall average thickness.

In all three of the tests, the epoxies cured and hardened on the crude oil. Figures 3-11 and 3-12 show the results of the three tests 90 min after mixing. The epoxy layers were hardened and bonded to the sides of the glass beakers. For the viscosity test, the epoxy also bonded to the aluminum piping. In flowing and curing, the epoxy apparently displaced the crude oil from the glass surfaces, resulting in a good bond.

Scaling laws do not permit the results of bench scale experiments to be extrapolated to behavior that might be expected in the raisebores or Service Shaft. The laws of similitude require scaling of not only geometry, but forces as well. These include inertia, gravity, surface tension, viscosity, and the exothermic reaction of the grout. Therefore, the above lab scale test results only demonstrate several important properties of the material (e.g., floating, spreading, leveling, hardening). The results do not prove that the epoxy will meet the requirements in the raisebores or Service Shaft. To better define large scale material behavior, the quarter scale test and pretests results are needed.

## **3.2 Quarter Scale Pretests**

### **3.2.1 Purpose of Pretests**

In preparation for the quarter scale test, several different scales of pours were made and instrumented to measure thermal buildup. Also, the

relative amounts of different ingredients were experimented with to test the sensitivity of the mix and improve its characteristics.

### **3.2.2 Pretest Procedures**

Of the many pretests performed, only a few examples are discussed here to illustrate some of the different undesirable results that one may find as a result of altering the the formulation. The scale included cup size up through 55 gal drum pours. The pours discussed here were on crude oil, although others were not. The epoxy was examined for evidence of density gradients, excessive thermal buildup, **gas** generation, and cracking. An undesirable reaction may exhibit density gradients, non-homogeneous expansion, cracking, etc. In general, these undesirable features are lessened as the exotherm decreases. Therefore, temperature was measured in most of the pretests with the goal of lowering the peak exotherm. An uncontrolled reaction would tend to produce high temperatures or excessive amounts of gas resulting in a foaming of the material. Uncontrolled or "runaway" reactions were not observed in any of the tests.

### **3.2.3 Pretest Results**

Each of the following tests involved a variation in the relative amounts of ingredients that goes into the basic formulation. The tests illustrate different undesirable results, with the exception of the final pretest discussed. That formulation was used in the quarter scale test.

Figure 3-13 shows two bucket scale pretests that exhibited a relatively large amount of gassing as evidenced by the bubbled surface. Two different formulations were tested. Both of the grouts were poured on top of crude oil and the center temperature of the grout was measured. The formulation of set 2 (on right) resulted in a higher peak temperature than set 1 (on left) and a slight bulging of the surface. The standpipe through each of the experiments is placed there to eliminate oil pressure buildup due to thermal expansion during heating in a confined area--a condition that cannot occur in the mine.

Figure 3-14 shows a 55 gal drum pour on crude oil. Although a large amount of gassing was not evident, the high peak exotherm (307°F) resulted in a boil on the surface. The temperature of the crude oil near the interface of the grout peaked at 103°F from an initial temperature of 78°F.

Figure 3-15 shows another 55 gal drum pour over oil using a different epoxy grout formulation. The peak exotherm (284°F) was lower than the above 55 gal drum pour. The peak oil temperature increased from its initial temperature of 83°F to 100°F. The material did not bulge at the surface, however it cracked as evidenced in the picture.

Figure 3-16 shows the final pretest performed and therefore the formulation used in the quarter scale test. The 2.5 ft square pour was 1 ft deep over crude oil. The peak exotherm in the grout was measured at 264°F after 95 min. Oil temperature increased from 69.4°F to 92.7°F. No bulging, cracking, or excessive gassing was noted and the exotherm was significantly lower from previous formulations (some of which reached as high as 350°F).

Figure 3-17 is a bar chart of the measured peak temperatures and times to peak for three different scales of pours of the formulation selected for the quarter scale test--a 55 gal drum pour, the 2.5 ft square pretest discussed above, and a small 7 in dia by 4 in deep pour. It should be noted that the time to peak exotherm and the peak exotherm reached will vary if the temperature at the start of the reaction is varied. A lack of sensitivity to test size is noted due to the high thermal insulating properties of the grout. This helps explain why the oil temperatures in the above experiments remained relatively low in comparison to the grout temperatures. Because the grout is a poor conductor of heat, size effects are negligible.

### 3.3 Quarter Scale Test

#### 3.3.1 Purpose of Quarter Scale

The purpose of the quarter scale material characterization test was to

demonstrate the following characteristic at a large scale: the ability of the grout to float on crude oil, spread, and encompass steel piping over a 12 ft flow distance, not adversely react either chemically or mechanically, and harden within a few hours.

### **3.3.2 Quarter Scale Test Procedures**

In order to demonstrate these qualities, a 3 ft wide by 12 ft long by 4 ft deep container with six 18 in dia steel pipes spaced at 2 ft was fabricated. Figure 3-18 shows a top and side view of the test configuration. This configuration simulates in full scale one row of pipes in the Service Shaft (see Figure 1-2). Thirty percent of the total shaft area and 38 percent of the piping in the shaft is represented. As currently planned, the grout would be introduced near the center of the Service Shaft, thus requiring it to flow a much shorter distance and surround fewer pipes along a given flow path than that used in this model. The flow requirements for the 6 ft dia raisebores (without pipes) are much less stringent than that required in the quarter scale test.

As illustrated in Figure 3-18, the pipes were positioned 3 in from each end, and the spacing varied 3, 6, and 9 in from the side wall of the container which was Lexon. The distances between the pipes and the wall was varied because of the results of Bench Scale Test 3 (which showed thinning of the epoxy layer in confined areas near the pipes). The other sides and bottom of the container were made of wood with a fiberglass lining. The container was initially filled with 12 in of water and 18 in of crude oil from Weeks Island. The water reduced the amount of crude oil needed and allowed a view on any precipitates from the oil. The bottoms of the pipes were perforated to prevent a buildup of fluid pressure due to heating. A water control line extended down into one of the pipes in order to maintain the initial fluid level if necessary. This control was not needed as the epoxy did not bond before its pour was complete. A 12 in layer of epoxy grout was dispensed over a time of approximately 90 min into the oil.

A low shear mixer and pneumatic powered piston pump was used to assure consistent batch characteristics. The epoxy flowed through a hose of approximately 2 in diameter into the end of the container from a drop of approximately 16 ft high. The flow rates from the pump and the exotherm were measured during the test. Thermocouples were placed at 4 in and 8 in into the top of the grout as well as 2 in and 9 in below the grout into the oil. After testing, full section thickness samples were taken at the inlet, middle, and maximum flow distance. The cores were sectioned and tested for density and glass transition temperatures. The glass transition temperature defines a well known property of polymers where the mechanical behavior of the material changes. Below the glass transition temperature the material is glassy or brittle; above it, the epoxy grout behaves rubbery and therefore has a lower modulus. To better simulate shaft conditions, the test materials were approximately 80°F at the time of test. A thermo-gravimetric test of the grout was performed to defined the temperature at which it starts to decompose or burn.

Because of the flammable nature of crude oil vapors, adequate ventilation was maintained and ignition sources were absent in the test area. Ventilation controls included both primary and secondary systems. The test container was capped with a glass, vapor proof lid and vented to the outside air. Ventilation over the entire test area was maintained with a large fan exhausting to the outside. Diking was constructed around the test area as a safeguard against accidental spill and sand was available for spill control and cleanup. The test box had multiple push-in plugs built for nozzling fire extinguishing chemicals into the box. One 150 lb and four 50 lb chemical fire extinguishers were available for fire suppression.

Figures 3-19 and 3-20 show top and side views of the test prior to pumping in the grout. The diking and vertical grout line are shown as well as the angled vent tube. Figure 3-21 shows an end view of the test along with the mixing and pumping equipment.

### 3.3.3 Quarter Scale Test Results

The test was performed on April 4, 1991. A VHS videotape of the experiment was made (Gravning, 1991b). Approximately eight pours were made over a 90 min period to form a layer of epoxy with an average thickness of 12 in.

Figure 3-22 shows a top view at the start of the initial pour. As seen from the side, Figure 3-23 shows the grout surrounding the first few pipes, A close up of the flow around the pipes is shown in Figure 3-24. These pictures were taken approximately 5 min into the test.

Twenty-one minutes after the pour was started, the entire surface area was covered. Figure 3-25 shows the initial layer that was formed. After 1 hr the layer thickness averages approximately 11 in as shown in Figure 3-26. The oil vortices in front of the pipes were caused by the edges of the epoxy entrapping some oil when surrounding the pipes. Rather than mixing in with the oil, the epoxy displaced the crude oil from the mix. As a result, the vortices are only superficial. A close up of a vortex is shown in Figure 3-27. A top view near the end of the test is shown in Figure 3-28. The surface is smooth and leveled. Variations in thickness along the length of the pour are due to a sloping underside.

Figure 3-29 shows the development of the epoxy layer at 20 min, 40 min, and 3.5 hrs after the start of the pour by plotting the location of the top and bottom of the epoxy layer with respect to the bottom of the test box. The Figure shows the tendency of the layer to level and become more uniform in thickness with time. Figure 3-30 plots the end thickness of the epoxy layer with time. The differences between end thicknesses are also plotted. In its final state, the difference in thickness was approximately 2.5 in over the 12 ft length with the top surface having leveled. Although not required, it is desirable that a flat level surface exist for the pouring of the next layer.

Figure 3-31 plots the volume of the epoxy layer with time. The slight

increases in volume after 90 min were mainly due to volumetric expansion of the grout due to its internal heating as pumping was complete by that time. Because the box confines the grout laterally, all expansion was vertical. This expansion was also noted in a 55 gal drum pour. In that test, the grout expanded approximately 10 percent in the vertical direction. In contrast, after cooling the grout shrank to form a hairline shrinkage crack between the container and the epoxy. In the quarter scale experiment, a shrinkage crack approximately  $1/16$  to  $1/8$  in wide formed after **cooldown** of the epoxy which translates into less than 1 percent shrinkage. Low shrinkage and high expansion are both desirable sealing characteristics.

Figure 3-32 shows the grout pump rate as estimated from the volume of the epoxy layer over time. The pump was rated at 8 gal/min. Pump rates varied considerably during the test--up to 12 gal/min. Although not shown in the Figure, pumping was temporarily stopped at times waiting on mixing of the grout. This lag time could have been eliminated by using a larger capacity mixer or one that can handle change out tubs. Similarly pump rates could be increased by using a larger pump. These options were not chosen for the experiment because they do not demonstrate worse case. By reducing the influx of grout, the momentum associated with the grout (a contributing factor to spreading) is reduced. Also, the overall cure state of the grout will be more mature (viscous) as it tries to flow around the piping.

The exotherms as measured at 4 and 8 in from the top of the grout layer and 2 in into the oil near the grout interface are shown in Figure 3-33. The thermocouples were located approximately 6 ft from each end and 9 in from the back (non-view) side on the test contained. The probes were located slightly off center from the width to reduce the effects of any heat sink caused by the steel pipes. The temperature peaked at 278°F in the epoxy and at 88°F in the oil. An additional temperature probe located at 9 in into the oil remained essentially unchanged during the experiment. The oil and grout temperatures were not high relative to those measured in the pretests, nor were any signs of excessive heat observed such as surface bulging or upheaval of the grout.

Figures 3-34 and 3-35 shows front and back views of the epoxy layer two days after the test with the test box removed. Figure 3-36 shows a single pipe after the epoxy layer was sectioned. These photos demonstrate leveling of the epoxy and bonding to steel. As a testimony to the strength of the material and its bonding, heavy sledgehammers were required to disintegrate and remove the epoxy surrounding the pipes.

Core samples were removed from the ends and middle of the epoxy layer, then sectioned and tested for density gradient and glass transition temperature (Rand and Montoya, 1991a). Figure 3-37 shows the density gradient across the thickness of the epoxy layer for each core. Core 1 was located at the grout inlet, Core 2 middle of the layer, and Core 3 at the far end. The Figure shows that density tends to increase toward the bottom of the layer and with distance from the grout inlet. Variations within these trends are due to variability within the different pours that were made. The overall average density was  $41.8 \text{ lb/ft}^3$  with a range from  $38.9$  to  $44.9 \text{ lb/ft}^3$ . These densities are significantly less than that of the oil ( $55 \text{ lb/ft}^3$ ). The trends indicate that longer, thicker pours are less desirable as maximum density increases.

The glass transition temperatures of the cores ranged from  $50$  to  $75^\circ\text{F}$ . On average, the transition from a glassy to a rubbery state was defined to onset at a temperature of approximately  $65^\circ\text{F}$ , where the shear modulus decreased by approximately two orders of magnitude from  $2 \times 10^5$  to  $3 \times 10^3$  psi at a temperature of approximately  $200^\circ\text{F}$  (Hance and Adolf, 1991). Figure 3-38 plots the shear modulus versus temperature. Because the temperature of the crude oil at Weeks Island is expected to be approximately  $80^\circ\text{F}$ , the grout once cooled to ambient temperature will exhibit a shear modulus of approximately  $7 \times 10^4$  psi. The mechanical behavior of the grout is farther evaluated in the mechanical tests performed in Chapter 4. The thermo-gravimetric analysis of the grout (burn test) showed the onset of decomposition at  $600^\circ\text{F}$ . This information is needed to predict the integrity of the epoxy seal in the event of an underground fire.

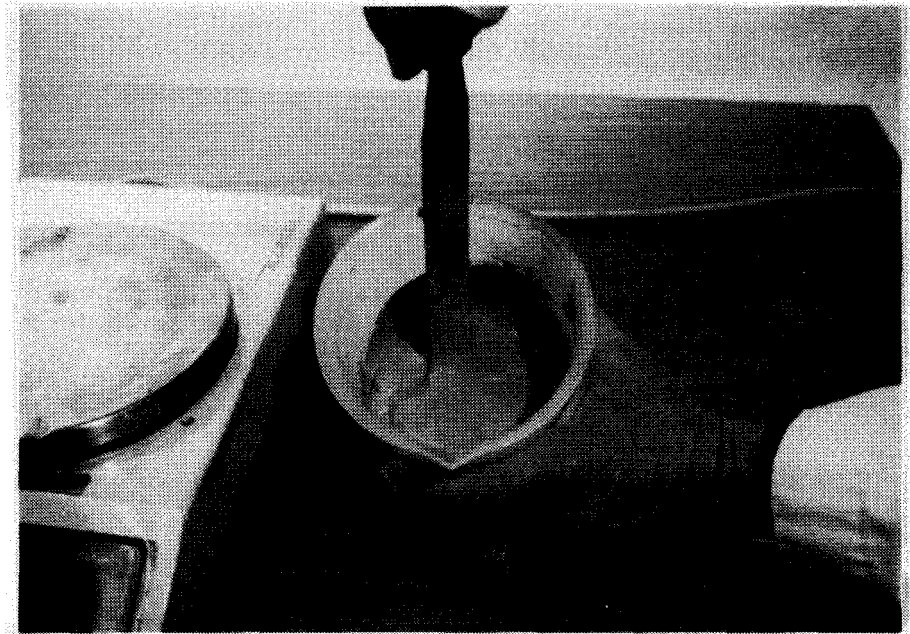


Figure 3-1. Mixing of Grout for Bench Scale Tests.

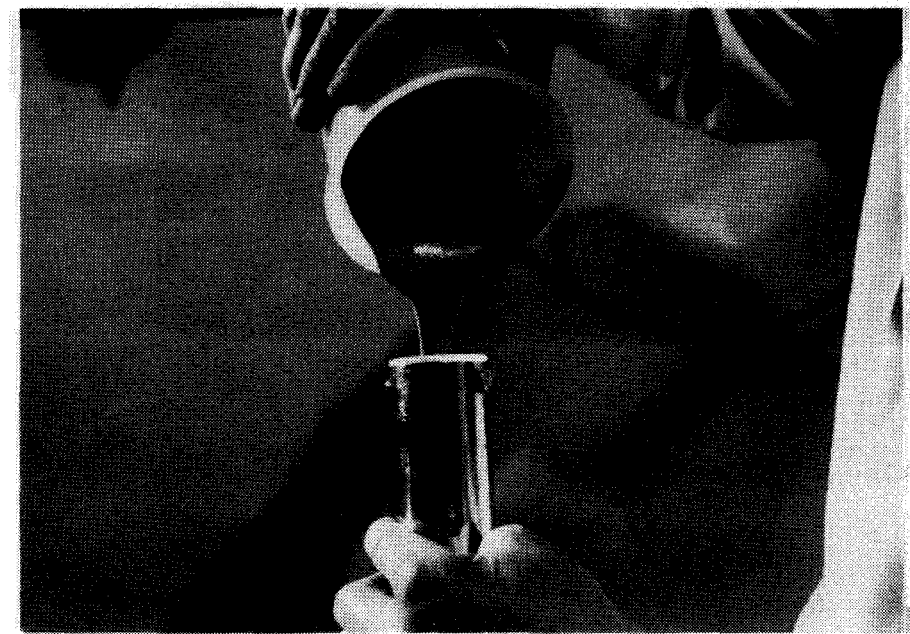


Figure 3-2. Pouring of Grout into Cemco for Bench Scale Tests.

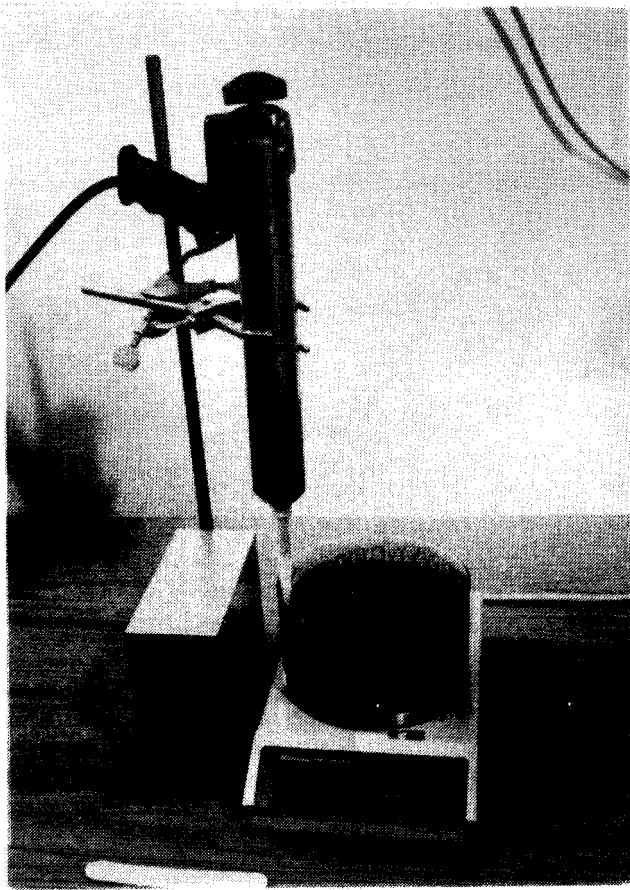


Figure 3-3. Experimental Setup  
for Bench Scale Tests.

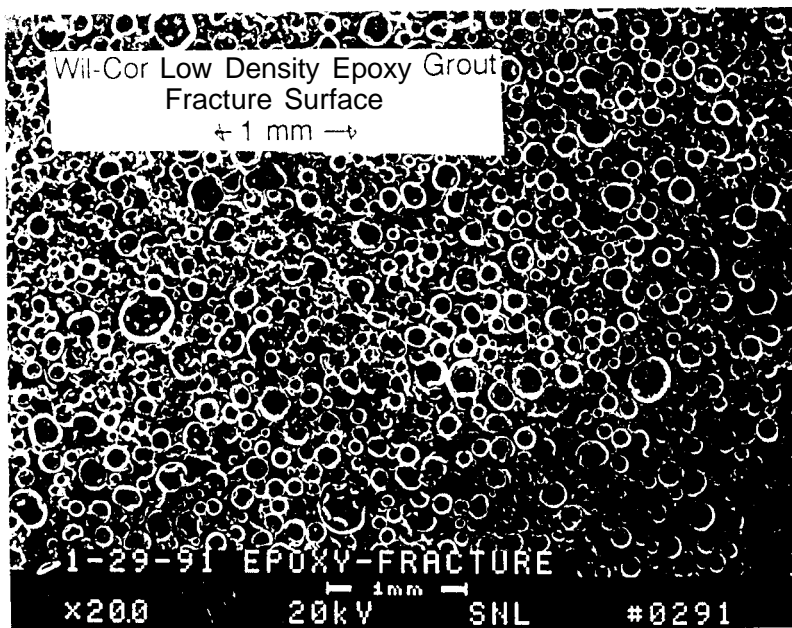


Figure 3-4. Electron Microscope Scan of Epoxy Surface at Low Power.

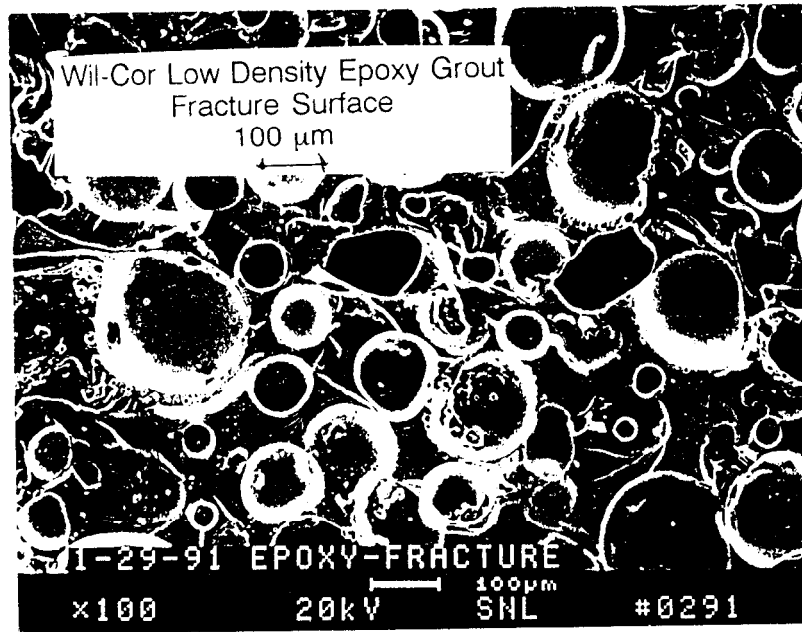


Figure 3-5. Electron Microscope Scan of Epoxy Surface at High Power.



Figure 3-6. Results of Float / Precipitates Test (5 min).



Figure 3-7. Results of Leveling Test (Top View at 5 min).

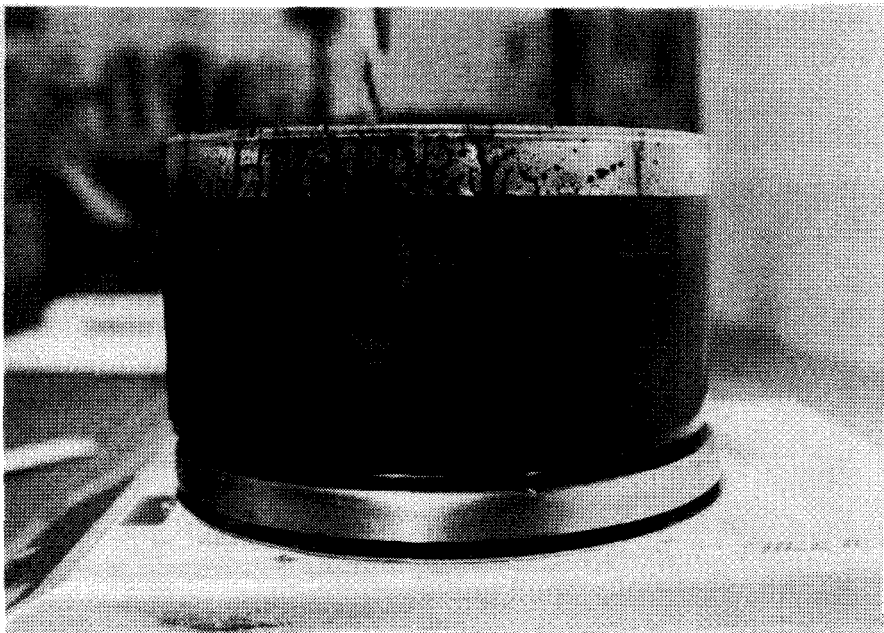


Figure 3-8. Results of Leveling Test (Side View at 5 min).

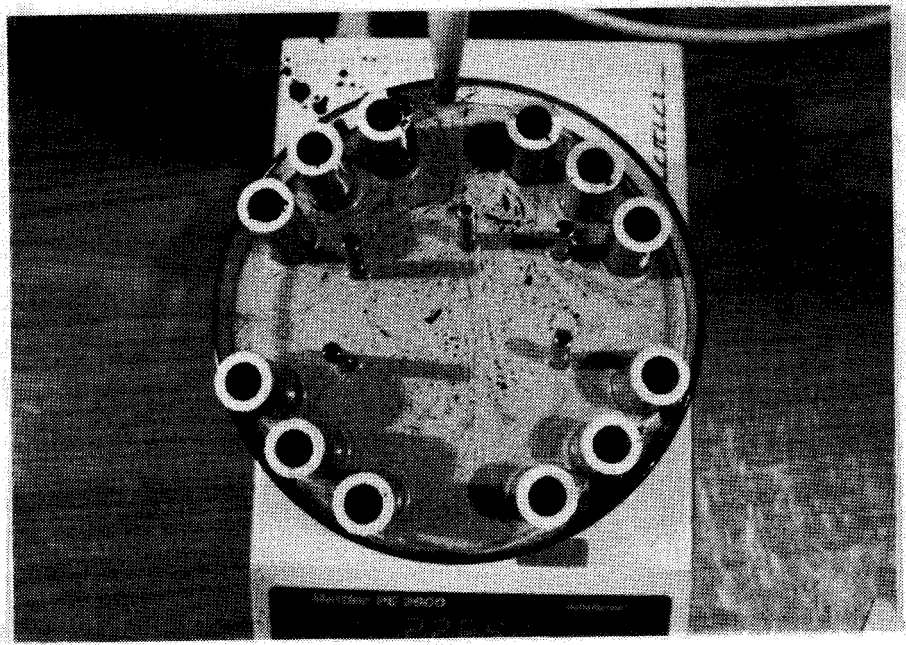


Figure 3-9. Results of Viscosity Test (Top View at 5 min).

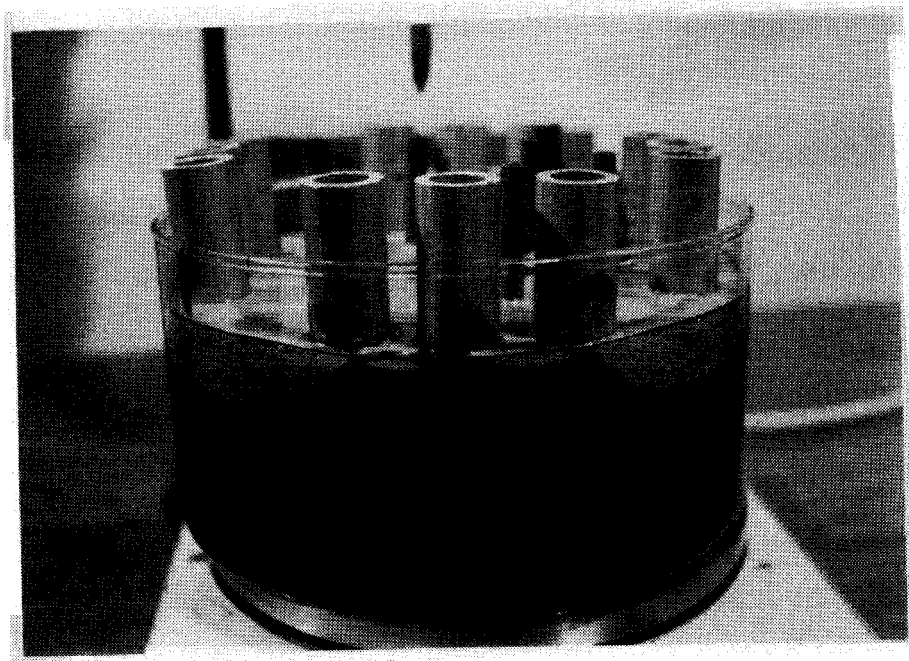


Figure 3-10. Results of Viscosity Test (Side View at 5 min).

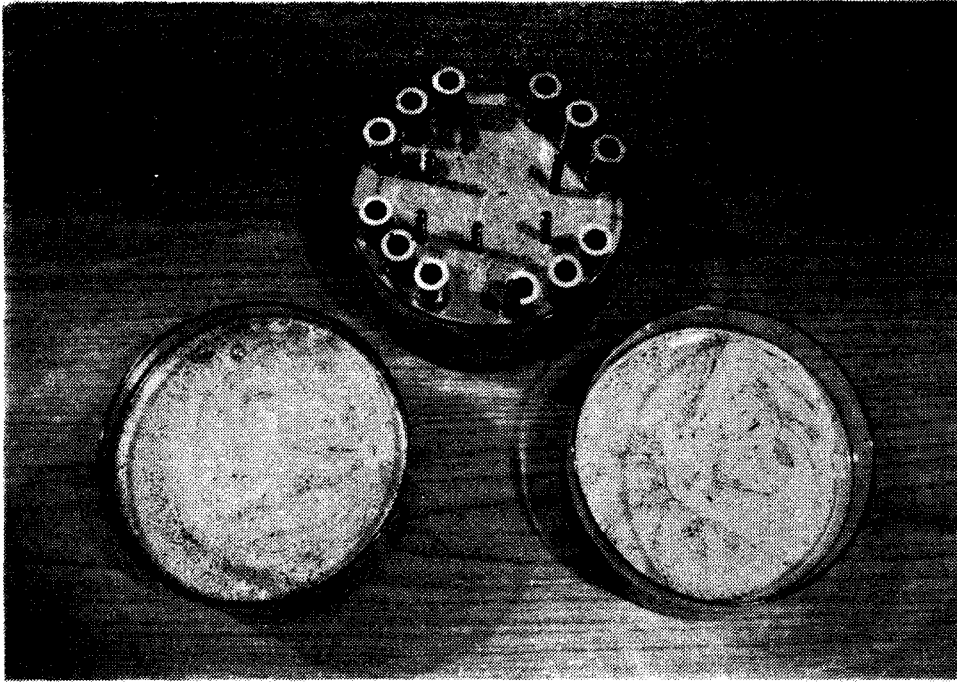


Figure 3-11. Results of Bench Scale Tests (Top View at 90 min).

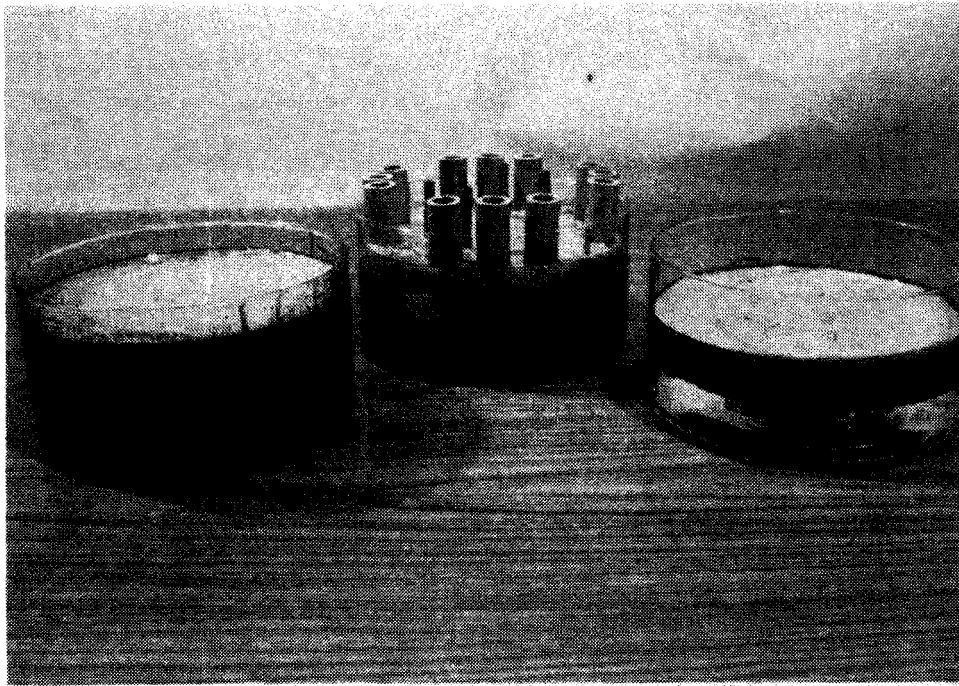


Figure 3-12. Results of Bench Scale Tests (Top View at 90 min).

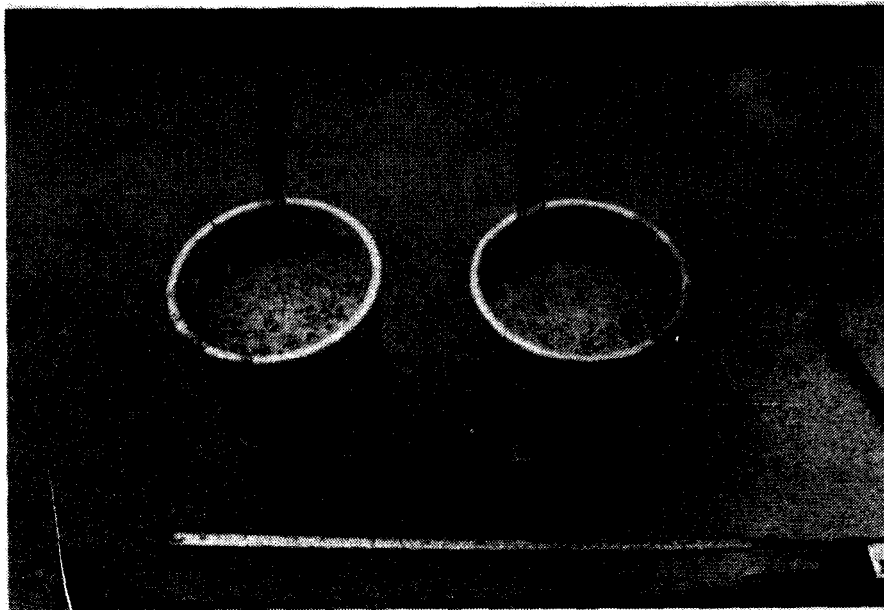


Figure 3-13. Bucket Scale Pre-Test Showing Gassing.



Figure 3-14. 55 Gal. Pre-Test  
with High Exotharm.

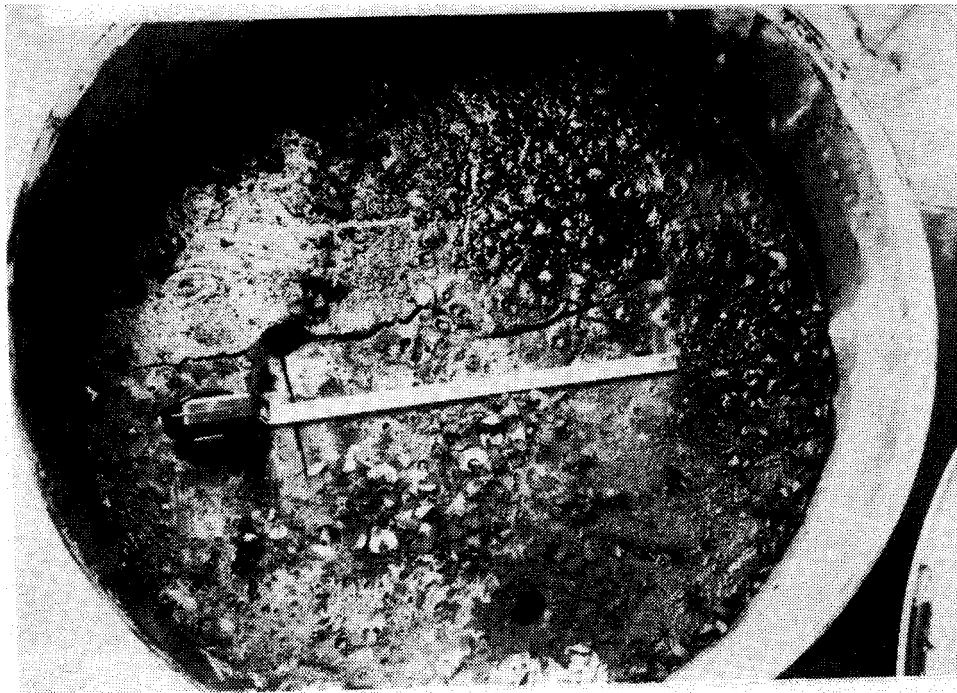


Figure 3-15. 55 Gal.Pre-Test Showing Cracking.



Figure 3-16. 2.5 x 2.5 ft. Pre-Test.

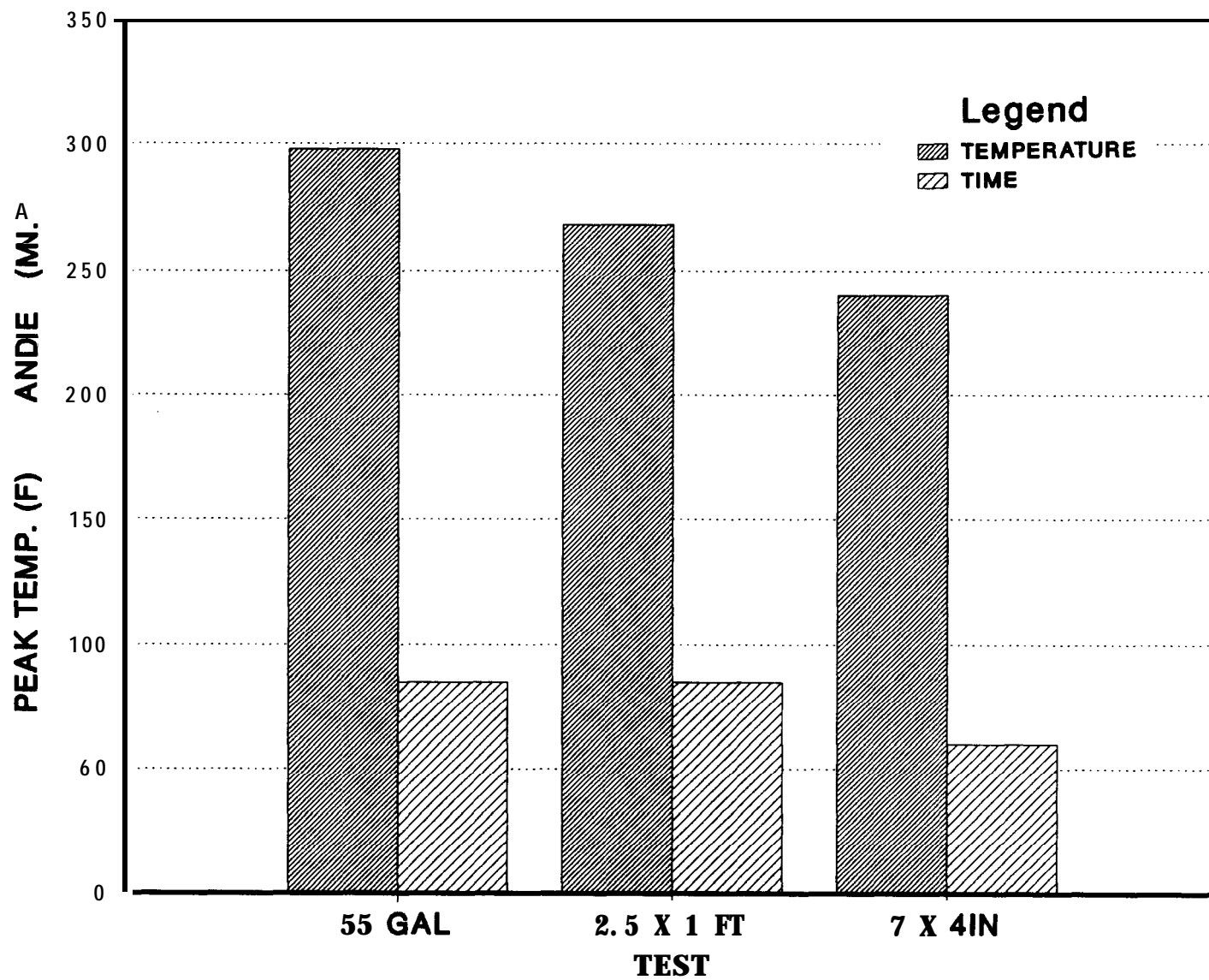


Figure 3-17. Effect of Pre-Test Size on Exotherm.

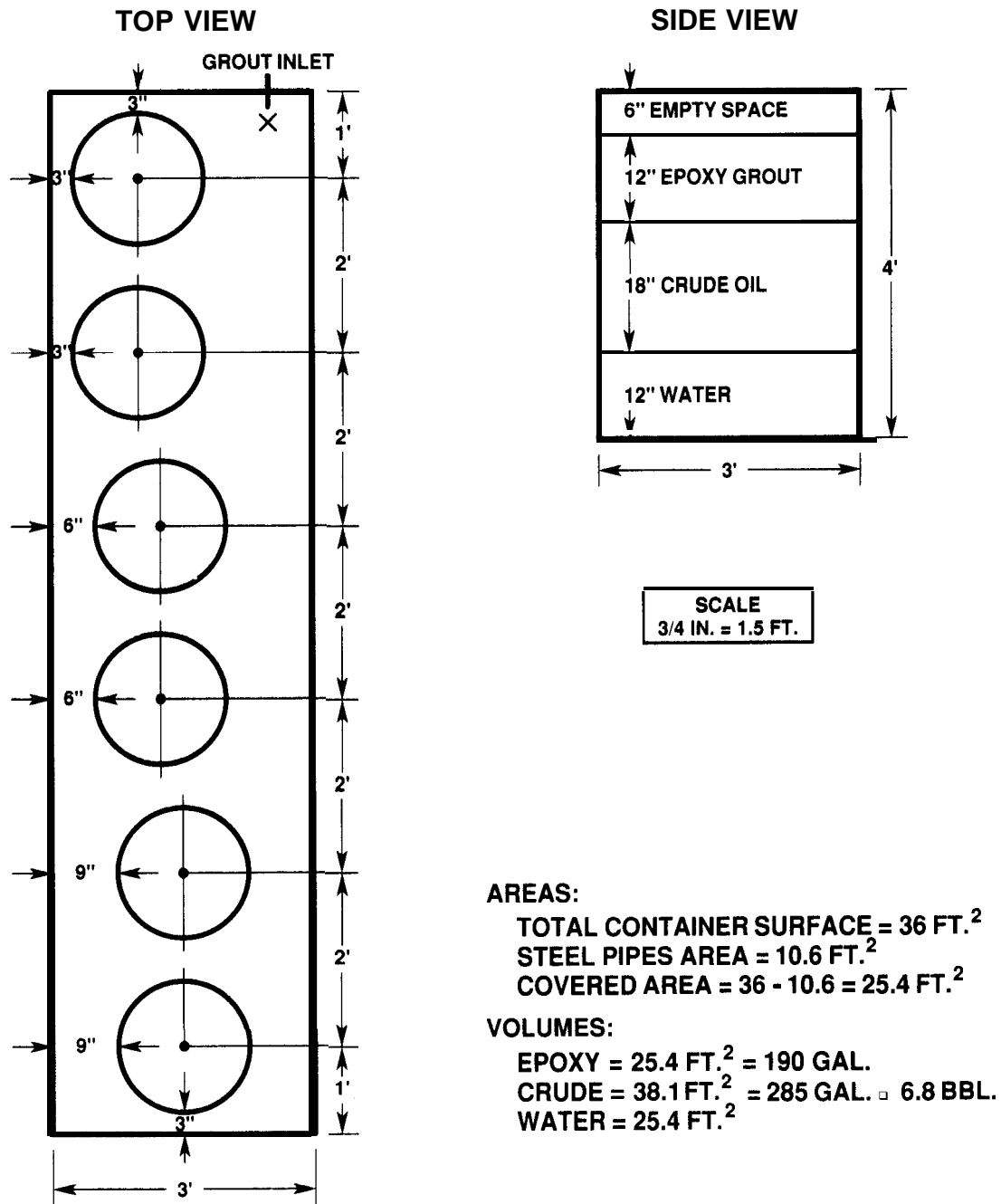


Figure 3- 18. Test Configuration for Quarter Scale.

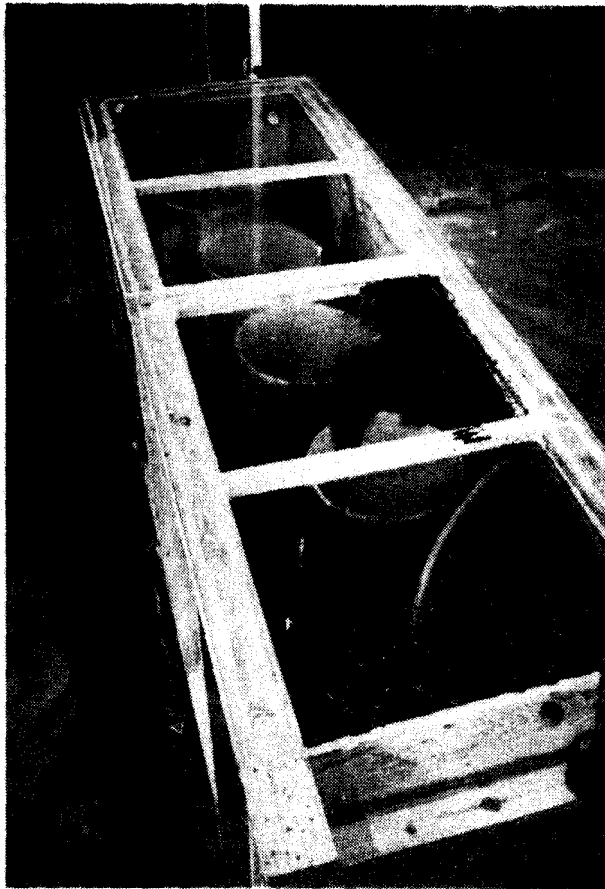


Figure 3-19. Top View of  
Quarter Scale.

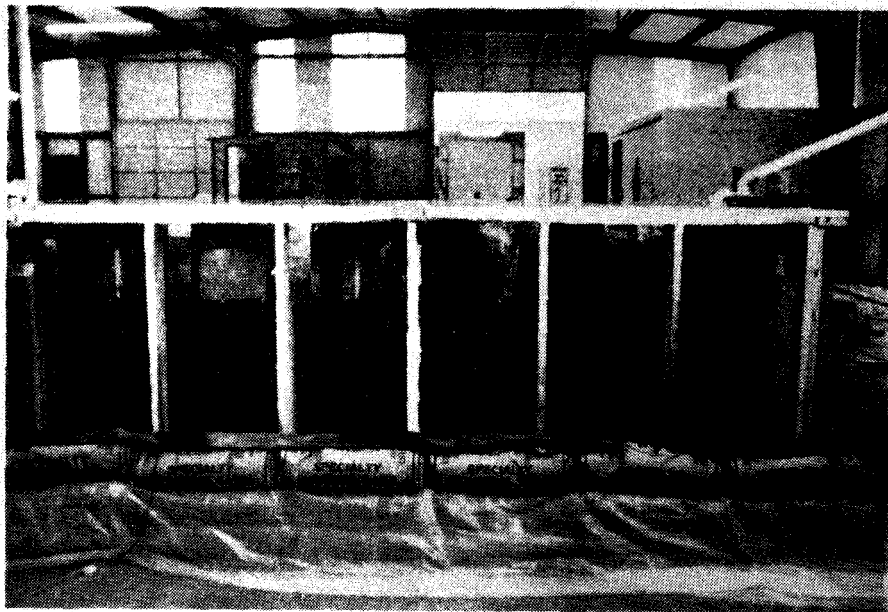


Figure 3-20. Side View of Quarter Scale.

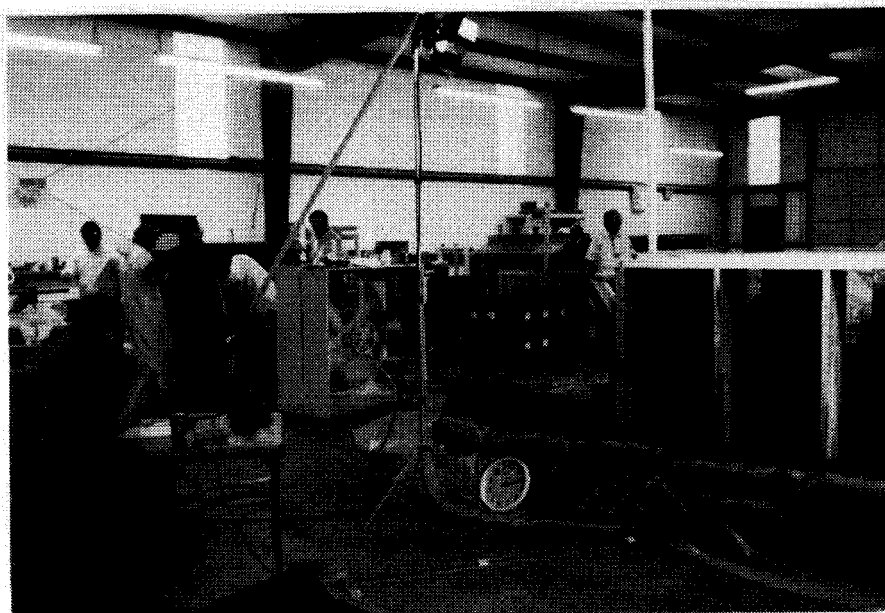


Figure 3-21. End View of Quarter Scale.

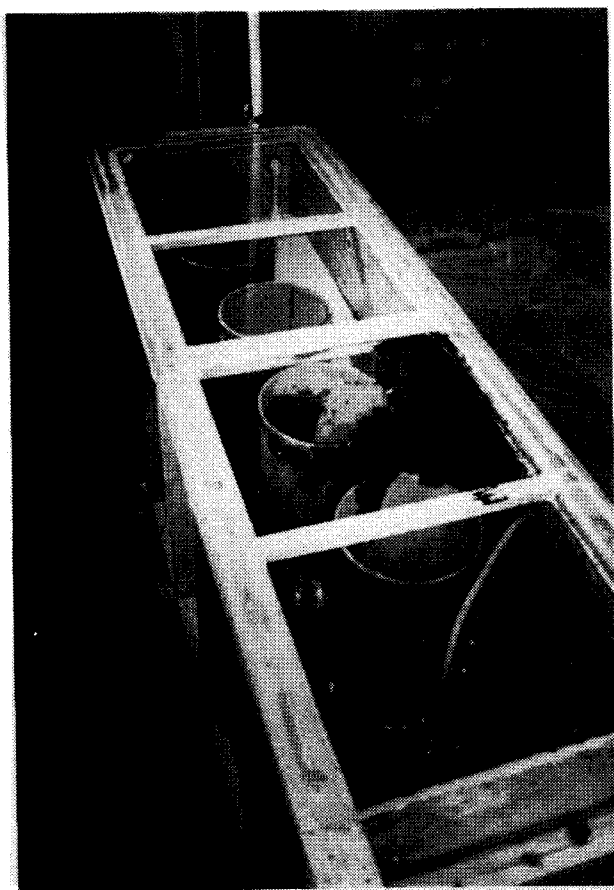


Figure 3-22. Top View of Quarter Scale at Start of Pour.

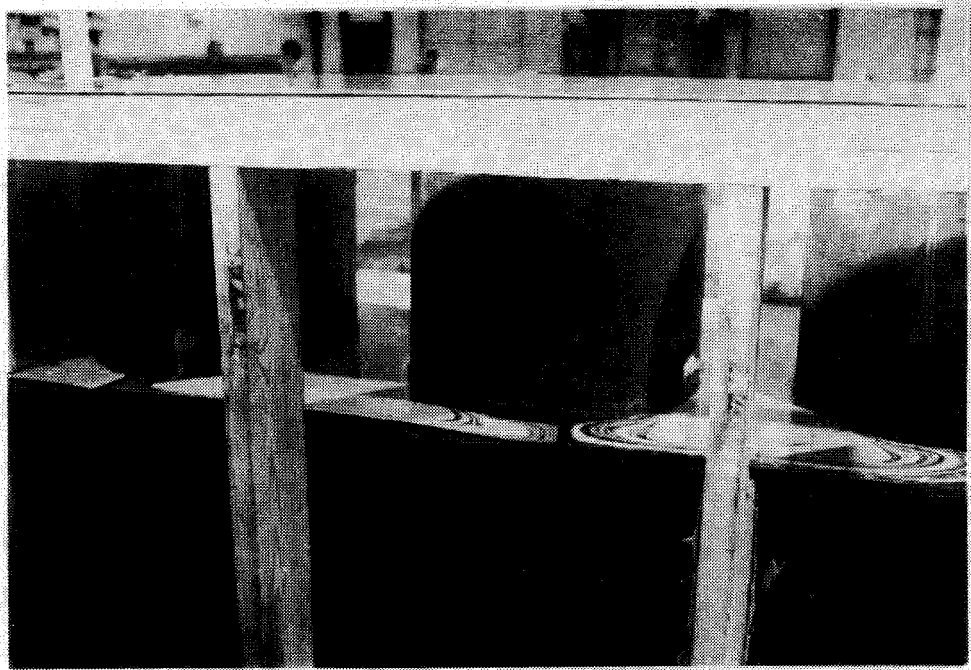


Figure 3-23. Side View of Quarter Scale at Start of Pour.



Figure 3-24. Close Up of Grout in Quarter Scale at Start of Pour.

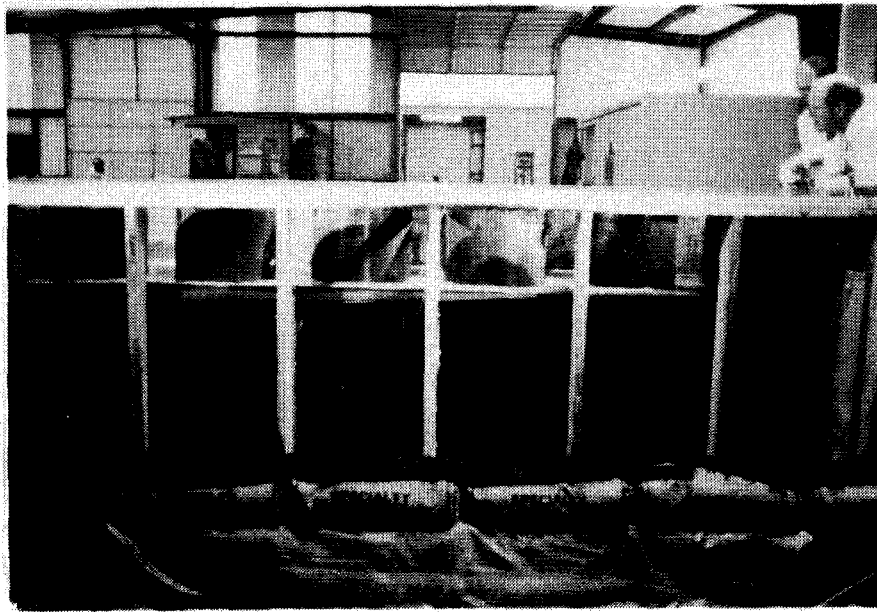


Figure 3-25. Initial Grout Layer Formed in Quarter Scale.

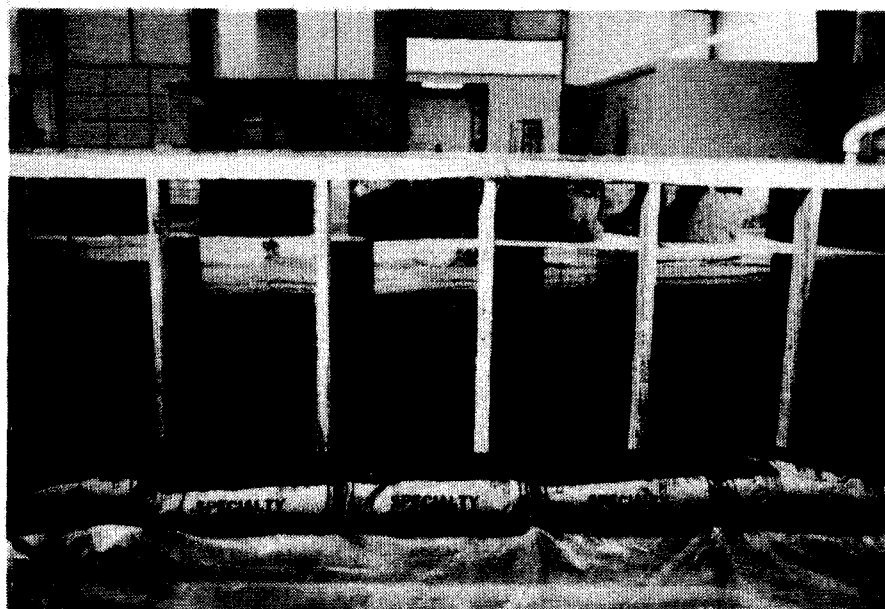


Figure 3-26. Grout Layer in Quarter Scale 1 Hour After Pour.

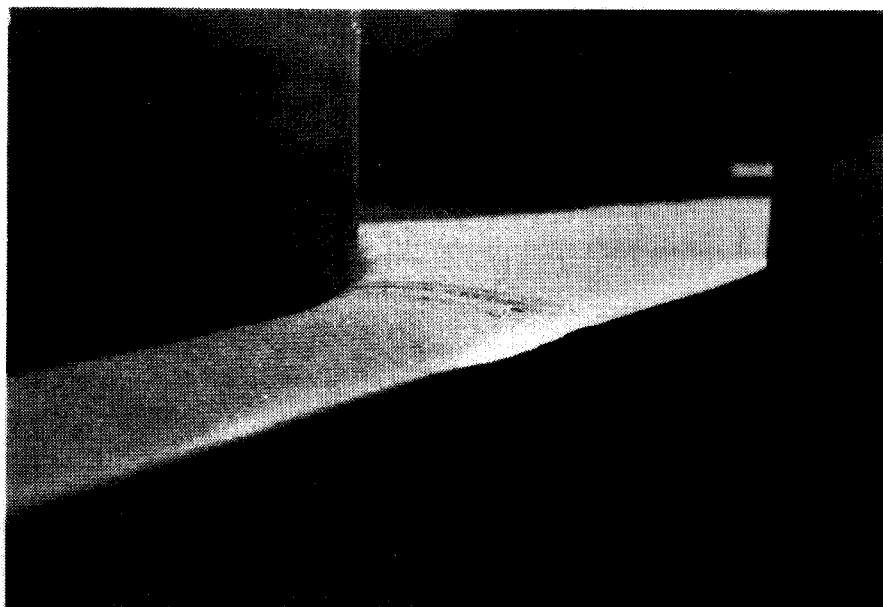


Figure 3-27. Close Up of Oil Vortex in Quarter Scale



Figure 3-28. Top View of Quarter Scale at End Near End of Test.

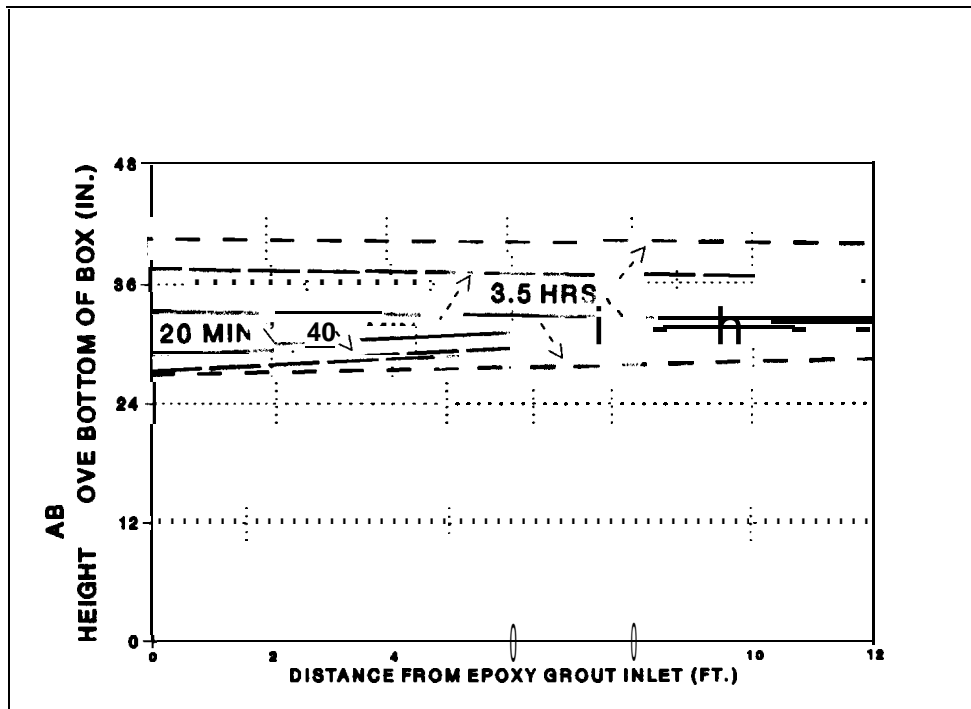


Figure 3-29. Development of Layer in Quarter Scale.

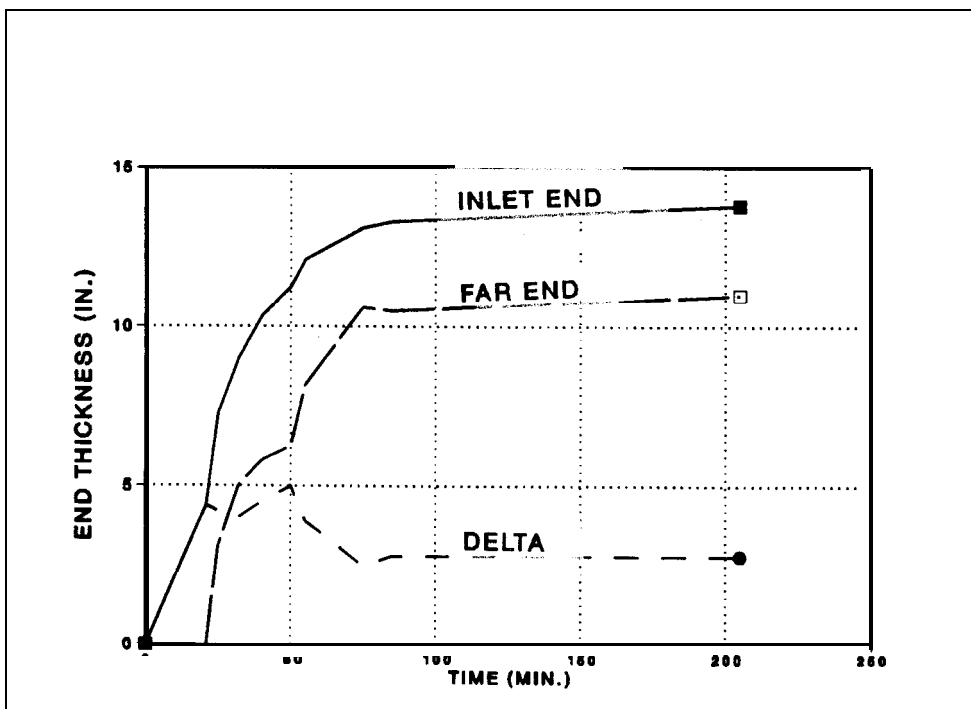


Figure 3-30. Measured End Thickness in Quarter Scale.

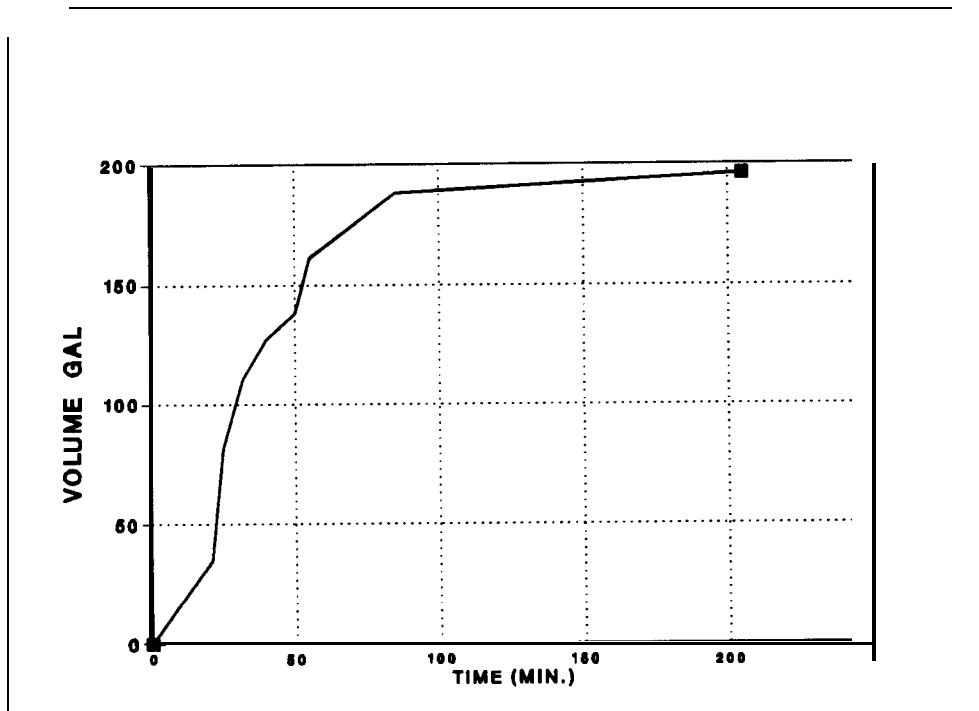


Figure 3-31. Volume of Grout Layer in Quarter Scale.

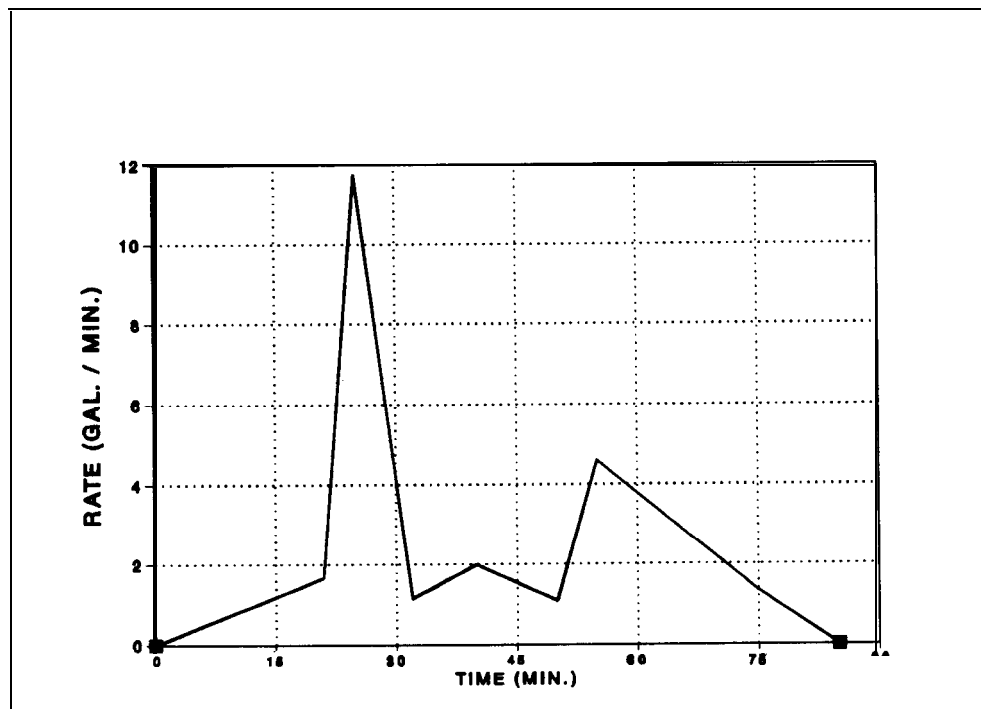


Figure 3-32. Pump Rates for Quarter Scale.

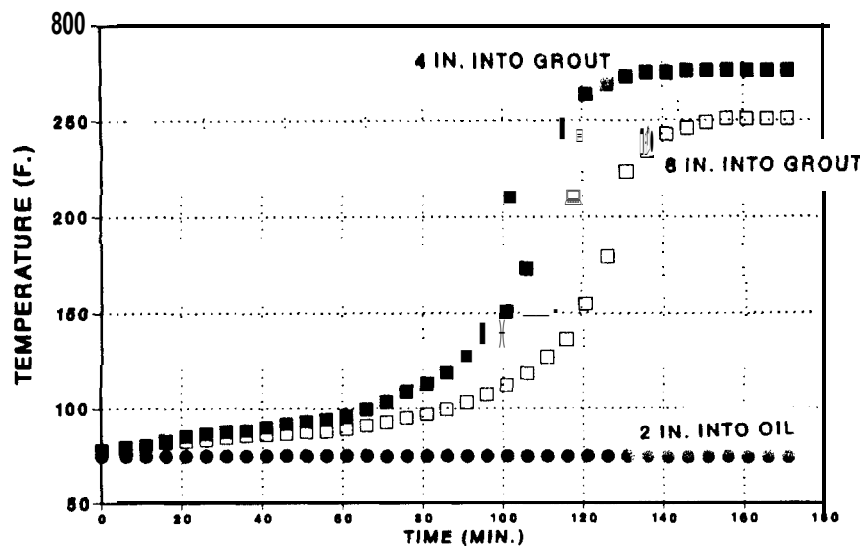


Figure 3-33. Exothermic History of Quarter Scale.



Figure 3-34. Front View of Quarter Scale at Disassembly.

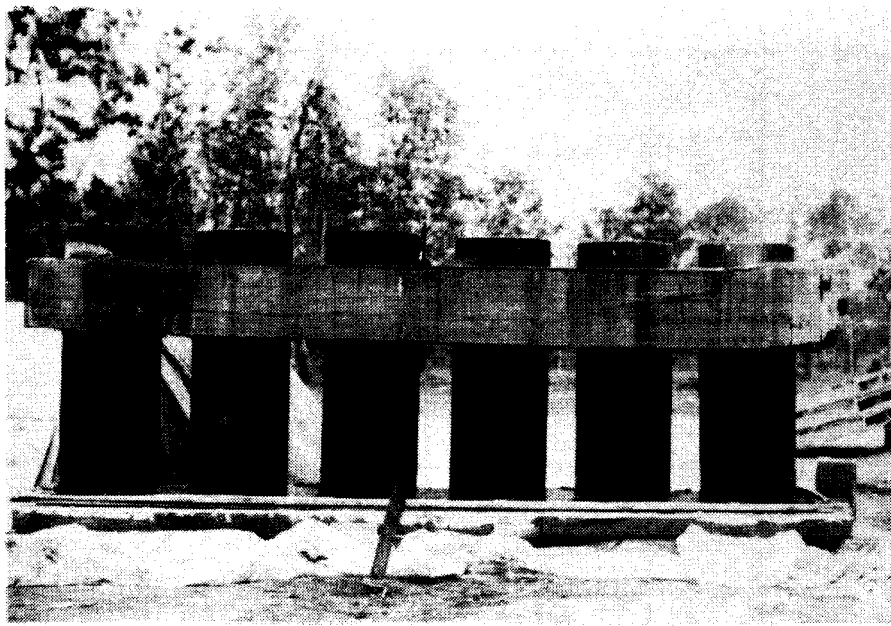


Figure 3-35. Rack View of Quarter Scale at Disassembly.

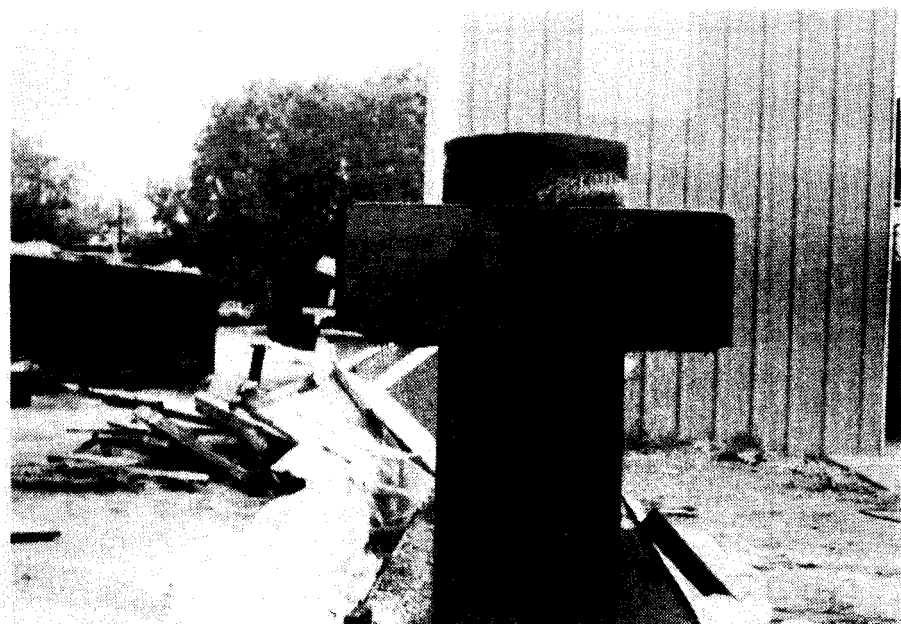


Figure 3-36 Isolated Pipe in Quarter Scale at Disassembly.

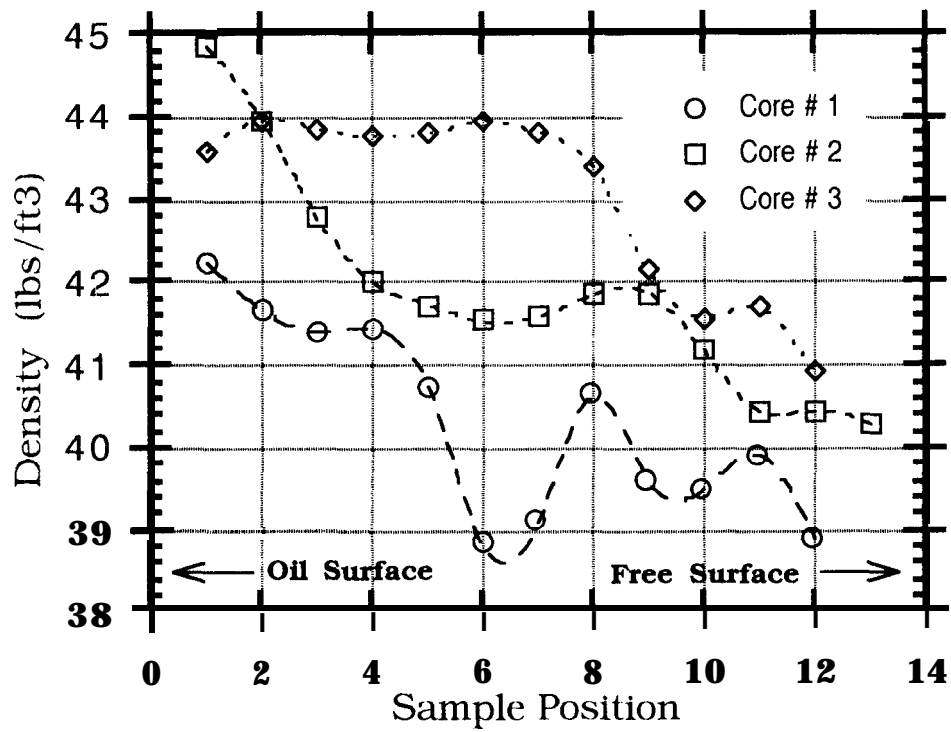


Figure 3-37. Distribution of Grout Density for Quarter Scale.

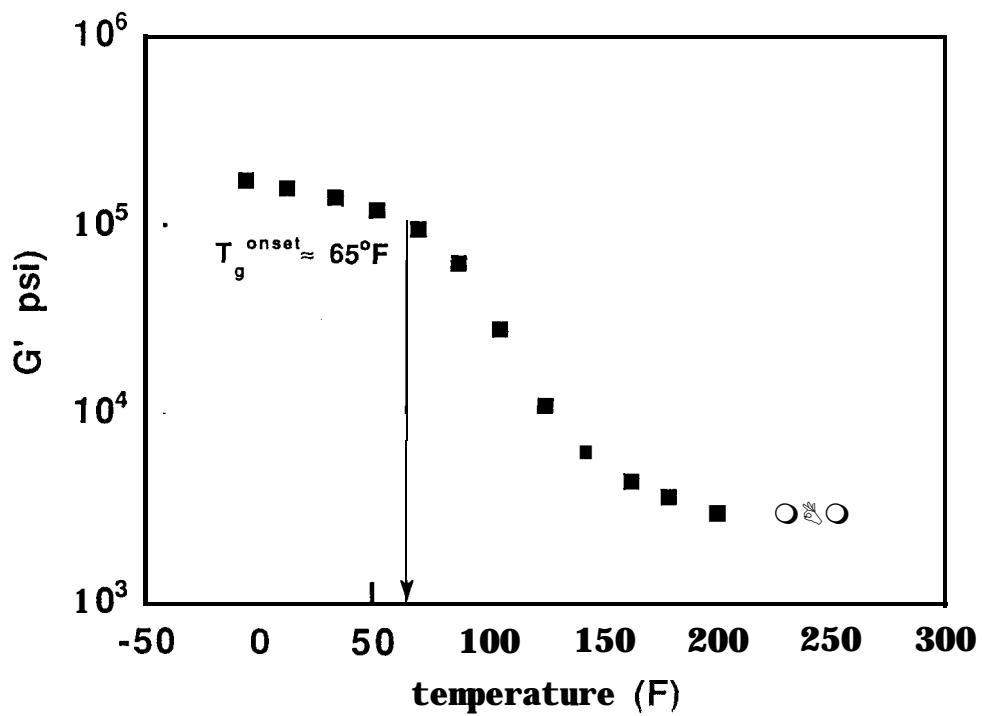


Figure 3-38. Temperature vs. Shear Modulus of Epoxy Grout.

## **4.0 MECHANICAL STRENGTH TESTS**

The structural stability of a low density epoxy grout plug and its ability to bond to the salt and pipes will primarily depend upon the shear strength at the epoxy/salt and epoxy/steel interfaces and the tensile strength of the material itself. These parameters result from treating the plug as a uniformly loaded plate, bonded at its edges and deformed in bending.

In order to characterize the strength parameters, both bond and tension tests were conducted. The cured age of the epoxy was varied (2 hrs, 6 hrs, and 24 hrs) as well as the interface conditions (crude oil wetted vs. dry) for the bond test. The wetness of the salt shaft wall and steel piping is unknown, but they may be coated with a film of crude oil. The purpose, procedures, and results for the bond tests and the tension tests are discussed in Sections 4.1 and 4.2, respectively.

### **4.1 Bond Strength to Salt and Steel**

#### **4.1.1 Purpose of Bond Tests**

The purpose of the tests was to determine the minimum interfacial shear strength of epoxy grout on steel and salt for both dry and wetted (crude oil) interface conditions over time.

#### **4.1.2 Bond Test Procedures**

Figure 4-1 shows the test configuration used to determine the minimum interface strength of epoxy grout bonded to salt and steel. A thin film of epoxy grout was placed between the steel cylinder and salt block and allowed to cure for a specified time. For wetted interfaces, the steel and salt were wiped with crude oil. The hollow cylinders with an inside diameter of 0.416 in and an outside diameter of 0.5 in were rotated 3.6 deg/min until the weakest interface or any of the material failed. Torque (in-lb) was measured and then converted to shear strength (psi) after multiplying torque by 9.8.

Table 4-1 shows the curing times and interface conditions for the shear tests. Two tests were performed for each test condition for a total of 12 shear tests.

**Table 4-1**  
**Test Matrix for Bond Strength Tests**

<b>Interface Condition</b>	<b>----- Curing Time -----</b>		
	<b>2 hrs</b>	<b>6 hrs</b>	<b>24 hrs</b>
dry	2d-1*	6d-1	24d-1
	2d-2	6d-2	24d-2
crude	2c-1	6c-1	24c-1
	2c-2	6c-2	24c-2

\*designation indicates sample age in hrs, interface condition (d=dry, c=crude), and sample number (two for each test condition).

The salt blocks and steel cylinders were preheated and specimens were cured at a constant temperature of 110°F. This temperature is an estimate of the average interfacial temperature expected in the Weeks Island Service Shaft. In the shaft, the thermal history at the interface will start at ambient (80°F), increase to a maximum temperature, and then cool to ambient.

For dry interface conditions the bottom surfaces of the steel cylinders were lightly abraded using a sand paper and then wiped with alcohol. The surfaces of the steel and salt block were blow dried using pressurized air. The epoxy resin and curing agents were mixed in equal parts by weight and the grout applied to bottom of cylinders. Only a small amount of grout was used to cover the cylinder bottoms with none allowed on the sides of cylinders. Thickness of epoxy film was as

uniform as possible for all specimens. The steel cylinders were rested on the center of the salt blocks and cured for the specified times to testing.

The crude oil interface samples were prepared as above, except the bottom surface of steel cylinders and entire surfaces of the salt were wiped with crude oil prior to applying any grout.

#### 4.1.3 Bond Test Results

Table 4-2 shows the measured shear strength (psi) of the samples and calculated averages for each sample tested. Sample identification numbers are in parenthesis.

Table 4-2  
Bond Test Results (psi)

Interface Condition	----- Curing Time -----			avg.
	2 hrs	6 hrs	24 hrs	
dry	988 (2d-1)	525 (6d-1)	1209 (24d-1)	907
	596 (2d-2)	729 (6d-2)	650 (24d-2)	658
crude	740 (2c-1)	1044 (6c-1)	751 (24c-1)	845
	754 (2c-2)	1074 (6c-2)	353 (24c-2)	727
avg.	770	843	741	784

The limited number of samples prevents a more rigorous statistical analyses other than the averages computed above. From the averages no significant differences are noted. The reason for this is the samples did not fail along either the epoxy/salt or the epoxy/steel interfaces

rather, the salt sheared. Therefore, the interfaces were stronger than the shear strength of the salt itself and the values in Table 4-2 represent the shear strength of the salt. Visual inspection of the samples showed the epoxy to displace the crude oil from the salt. This may help to explain why good bonding was achieved even when both the steel and salt were initially coated with crude oil.

The salt and steel interfaces showed no signs of material incompatibilities, such as corrosion. It is expected that the epoxy will inhibit pipe corrosion and thus benefit the oil withdrawal system.

## 4.2 Grout Strength and Modulus

### 4.2.1 Purpose of Strength Tests

The purpose of the testing was to determine stress-strain relationship and ultimate strength in tension of the epoxy grout cured at various times.

### 4.2.2 Strength Test Procedures

Figure 4-2 shows the test configuration used to determine the tensile strength of the epoxy. The stress-strain profiles were obtained for each test from the displacements measured by a linear variable differential transformer (LVDT) placed across the failure plane. Using the initial linear portion of the stress-strain data, the elastic modulus of the material was calculated. The samples were pulled in tension at a crosshead speed of 0.05 in/min and the load (lb) recorded. This value was converted to stress by dividing by the cross-sectional areas of the sample ( $.102 \text{ in}^2$ ).

Table 4-3 shows the curing times for the tension tests. Two tests were performed for each test condition for a total of six tension tests.

**Table 4-3**  
**Test Matrix for Tension Tests**

---

	CuringTime -----		
	2 hrs	6 hrs	24 hrs
	2-1*	6-1	24-1
	2-2	6-2	24-2

---

\*designation indicates sample age in hrs and sample number (two for each test condition).

---

Similar to shear tests, the tension specimens were cured at 110°F. This temperature is intended to represent the average grout temperature along the lower surface of the epoxy plug. This location reflects the highest tensile stresses that may be expected due to bending of the plug. The mold used to cast the samples was preheated in each case. A graphite based mold release compound was used to ease specimen removal from the mold after curing.

The specimens were cast into a dog bone shape and allowed to cure to the times specified above in the test matrix.

#### **4.2.3 Strength Test Results**

Figure 4-3 shows the stress-strain profile of each sample. The 2 hr samples showed a lower strength and modulus than the 6 and 24 hr samples. No significant differences are noted in strength between the 6 and 24 hr samples. This is probably true of modulus as well, because sample 24-1 was slightly warped which can explain its lower modulus relative to sample 24-2 and the 6 hr samples. The preyield modulus (obtained from linear portion of curves) and ultimate tensile strengths are listed in Table 4-4.

**Table 4-4**  
**Tension Test Results (psi)**

---

	----- Curing Time -----		
	2 hrs	6 hrs	24 hrs
Sample ID	2-1	6-1	24-1"
Strength	1620	2060	2160
Modulus	2.90 x 10 <sup>5</sup>	4.01 x 10 <sup>5</sup>	2.90 x 10 <sup>5</sup>
Sample ID	2-2	6-2	24-2
Strength	1520	2160	2110
Modulus	1.17 x 10 <sup>5</sup>	3.90 x 10 <sup>5</sup>	3.65 x 10 <sup>5</sup>
avg. Strength	1570	2110	2135
avg. Modulus	2.04 x 10 <sup>5</sup>	3.96 x 10 <sup>5</sup>	3.65 x 10 <sup>5</sup>

\*sample slightly warped, modulus value not used in computing avg.

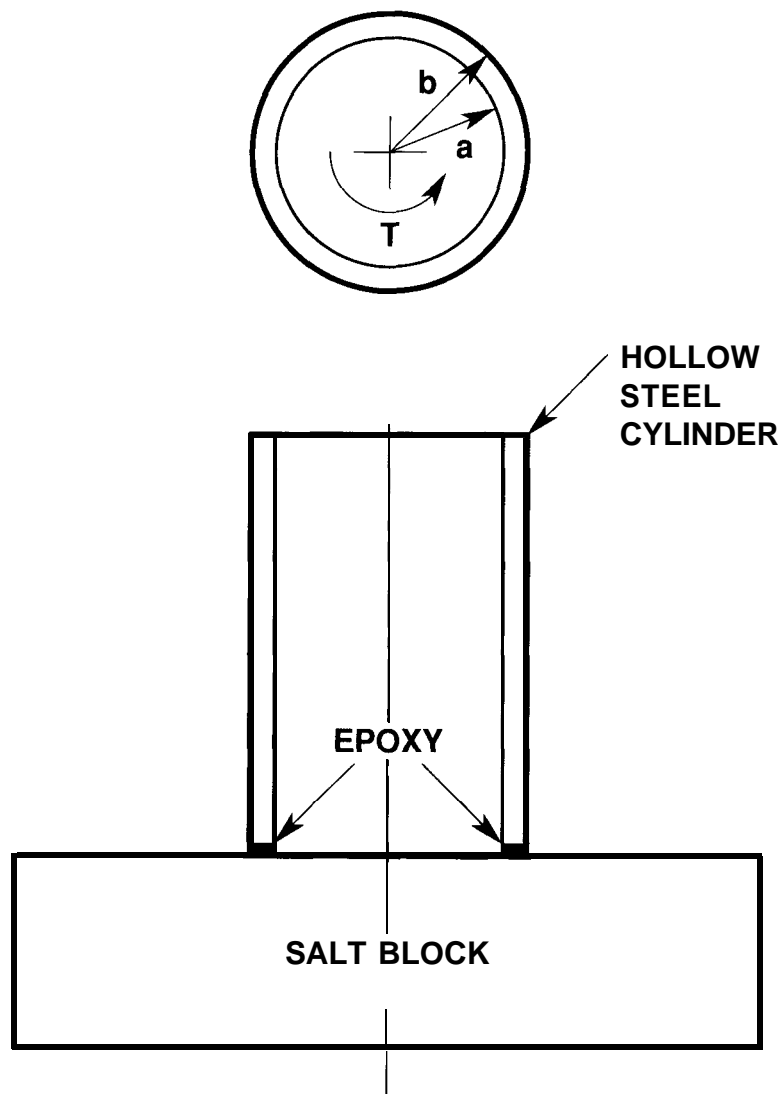
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The epoxy grout tested at its lowest tensile strength and modulus at 2 hrs. This is expected as the strength and modulus of epoxy increases with time. The decrease in modulus from 6 to 24 hrs is most likely an **artifact** of the testing. Statistical comparisons are meaningless because of the small number of samples used, therefore the results are at best, approximations of the true strengths of the grout. To measure any significant increases in strength or modulus beyond 6 hrs, will probably require a much longer cure time than 24 hrs, perhaps on the order of a week.

The strength of the epoxy targeted for use in the Service Shaft and raisebores may vary considerably from the above results due to the increased exothermic reaction of a larger mass of epoxy mass; however,

because the measured strengths are far above the 10 psi requirement (see Chapter 2), additional bond and strength testing was not pursued.

## SHEAR ANNULUS



$$\begin{aligned} T &= \text{TORQUE (in-lb)} \\ a &\approx 0.416 \text{ inch} \\ b &\approx 0.500 \text{ inch} \\ T(r) &= 2Tr/3.14 (b^4 - a^4) \\ T_{\max} &= 9.80 T_{\max} \text{ (PSI)} \end{aligned}$$

Figure 4- 1. Bond Test Configuration.

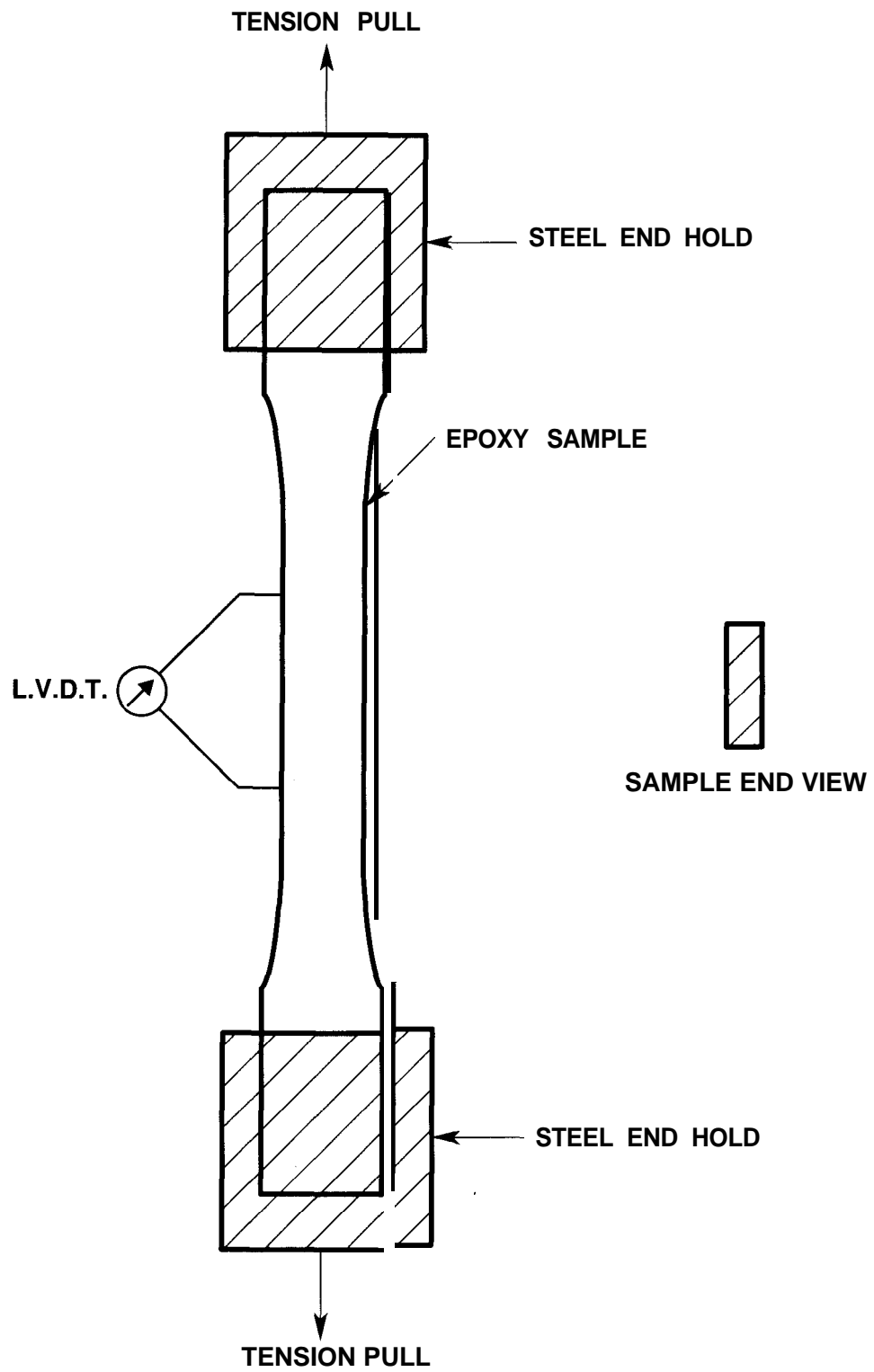


Figure 4-2. Tension Test Configuration.

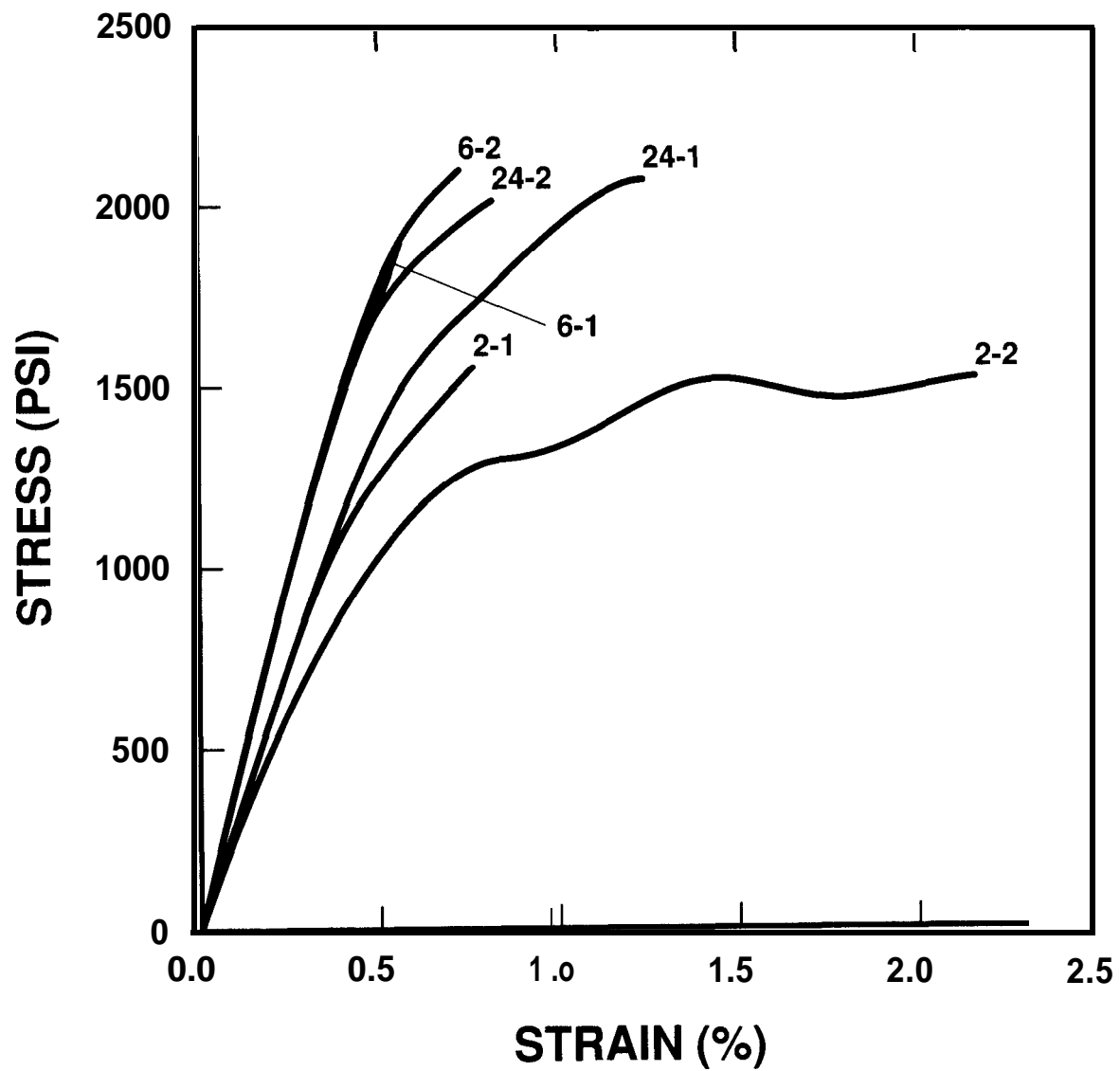


Figure 4-3. Stress - Strain Profiles for Tension Tests.

## 5.0 CONCLUSIONS

A low density epoxy grout was characterized using bench and large scale flow tests and mechanical tests. The material tested was Armor Plate Ultra Low Density Epoxy Grout 1000 available from Wil-Cor, Inc., of Pasadena, Texas.

The material is qualified for use in the Service Shaft and raisebores at the Weeks Island US Strategic Petroleum Reserve, Louisiana, on the basis of having demonstrated through testing the characteristics necessary to meet the requirements defined in Chapter 2. Compliance with each requirement is discussed below.

1. The grout was mixed, pumped, and delivered down to an oil surface without altering its characteristics. This was demonstrated in the quarter scale test which used a delivery system typical to that which may be used at Weeks Island.
2. The grout had a specific gravity less than 45 lb/ft<sup>3</sup> and floated on top of Weeks Island crude oil (55 lb/ft<sup>3</sup>). Density gradients measured throughout the quarter scale pour varied from 39 to slightly less than 45 lb/ft<sup>3</sup>.
3. The grout was not adversely affected by the crude oil. The resin, filler or aggregate, or any other component of the grout did not separate from the grout and sink into crude oil. Crude oil did not destabilize the mix and cause any material to sink into the crude oil. No adverse reactions with crude oil were noted in any of the tests performed.
4. The grout had a fluidity that allows it to spread across the surface of the crude oil from the injection point and flow around simulated vertical pipes in the Service Shaft, self-level, and completely cover the entire oil surface. The quarter scale configuration simulated in full scale one row of pipes in the Service Shaft. It represents the highest density of piping and the maximum expected flow distance for the grout.

5. The grout hardened within 24 hrs of the pour and was ready to accept the next grout (low or high density) pour. The epoxy layer in the quarter scale test had a hard level upper surface. The hardness of the grout was directly measured as modulus in the mechanical testing of the grout.
6. The grout developed a minimum shear bond strength to salt and steel well in excess of 10 psi 24 hrs after mixing for both clean dry and oil coated interface conditions. Minimum bond strengths were measured as greater than the shear strength of the salt itself (approximately 800 psi). The difficulty of dismantling the quarter scale test was a testimony to its bond strength.
7. The grout developed tensile strength well in excess of 10 psi 24 hr after mixing. Tensile strengths were tested at approximately 2100 psi.
8. The grout did not shrink more than 1.0 percent in a 24 hr period or develop any cracks. The grout in the quarter scale experiment shrank only 0.2 percent over its length and no cracks were evident.
9. The grout was formulated such that "runaway" or uncontrolled reactions did not occur. Uncontrolled reactions were not observed in any of the tests.
10. The grout did not cause temperature of crude oil to exceed 150°F due to the heat released from the epoxy. Crude oil temperatures remained below 100°F in most experiments.
11. The grout parts or packages had a shelf life of at least two weeks from date of manufacture.

To help assure performance of the grout materials and its delivery system at Weeks Island, a construction specification is provided in the attached Appendix. The construction specification is an example that reflects the recommendations of the author and it is not necessarily the one that will be approved and issued for construction.

The specification recommends that the raisebores be done prior to the Service Shaft--the more critical application. It defines quality control tests (Rand and Montoya, 1991b) to assure the low density grout used **downhole** is the same as that used in the qualification tests. Finally, the specification defines a test that will verify the permeability of the low density epoxy grout plug before the non-buoyant high density grout is poured.

## REFERENCES

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## APPENDIX

### EXAMPLE OF A CONSTRUCTION SPECIFICATION FOR LOW DENSITY EPOXY GROUT

#### PART 1 - GENERAL

##### 1.01 DESCRIPTION

- A. Work Included: Provide all labor, tools, and materials required to install a Low Density Epoxy Grout Plug in Raisebores No. 1 and No. 2, and subsequently in the Service Shaft where shown on the Drawings, as specified herein, and as needed for a complete and proper installation.
- B. Related Work:
  - 1. Documents affecting the work of this Section include, but are not limited to, other specifications related to Raisebores No. 1 and 2, Service Shaft, High Density Epoxy Grout, and Inflatable Packers.

##### 1.02 REFERENCES

The latest published version of Codes, Specifications, and Standards referred to by number or title shall form part of this specification. These publications are not furnished with the Contract Documents.

American Concrete Institute (ACI)

ACI 548.1R                      Guide for the Use of Polymers in Concrete

American Society of Testing Material (ASTM)

ASTM D638                      Tensile Properties of Plastics

##### 1.03 DEFINITIONS

- A. The Low Density Epoxy Grout as defined herein is a material that will float on crude oil and that meets or exceeds the physical characteristics and requirements identified in Part 2 "Materials". The light weight polymer material that will be placed by the CONTRACTOR on the crude oil surface and will provide support to and fully contain (prevent passage to oil) the pour of non-buoyant High Density Epoxy Grout.
- B. The High Density Epoxy Grout as defined herein is a **non-buoyant** material placed on top of the Low Density Epoxy Grout plug to provide structural and sealing properties in the raisebores and Service Shaft.

#### 1.04 SYSTEM DESCRIPTION

- A. The CONTRACTOR will supply and install a low density plug as shown on the Drawings and as specified herein. The plug material will be placed by CONTRACTOR on the crude oil surface. This plug will serve as a platform to support and contain the material used to form the first overlying layer of high density epoxy grout.
- B. The epoxy plug's function is to form an impervious barrier to the passage, or leakage, of a non-buoyant high density epoxy grout pour placed on top of the low density plug. It shall support the weight of the initial layer of high density epoxy grout plug until the high density grout plug sets and develops full strength.

#### 1.05 DELIVERY, STORAGE, AND HANDLING

The epoxy grout to be used in this project shall be delivered as a prepackaged, multi-part system. These components shall be delivered to the jobsite in original unopened packages, clearly labeled, with each batch identified. The grout components shall be stored at 70" 4 20°F in a dry environment. (Refer to Section 2.03.)

#### 1.06 SUBMITTALS

- A. Submit all items in accordance with the requirements of Section 01300, "Submittals." The Contract Submittal Requirements Listing (CSRL) in Section 01300 is a summary of all technical submittals and contains the items listed below. The list in this Section takes precedence over the CSRL.
- B. Submit the following to qualify the low density grout material at time of bid. Qualification tests will be performed at no cost to the GOVERNMENT. A large scale qualification test (Refer to Chapter 3.3 of this Report entitled "Quarter Scale Test, specifically Sections 3.3.1 "Purpose" and 3.3.2 "Procedures") is required to demonstrate the ability of the grout to: flow across a 12 ft long container of crude oil with six 18 in diameter steel pipes spaced at 2 ft intervals, cover the entire surface area, self level, harden and bond to the steel pipes resulting in a 1 ft thick layer of grout. The large scale test must use a mixing, pumping, and delivery system typical of that proposed for use at Weeks Island.
  - 1. Grout Plug Material Specification and Safety Data Sheets.
  - 2. Grout Physical, Thermal, and Chemical Properties for:

- a. Density of grout material.
  - b. Inertness to oil.
  - c. Spreading factor (refer to Attachment 4.01).
  - d. Peak exotherm and time to peak temperature.
  - e. Bond strength to salt and steel at 2 hr, 6 hr, and 24 hr cure for dry and crude wetted surfaces. (For example of a Test, refer to Chapter 4.1 of this report entitled "Bond Strength to Salt and Steel," specifically Sections 4.1.1 "Purpose" and 4.1.2 "Test Procedures.")
  - f. Tensile Strength, at 2 hr, 6 hr, and 24 hr cure. (For example of a Test Specification, refer to Chapter 4.2 of this report entitled "Grout Strength and Modulus", specifically Sections 4.2.1 "Purpose" and 4.2.2 "Test Procedures.")
  - g. Expansion during curing and shrinkage during cooling.
  - h. Glass transition temperature.
  - i. Flammability or hazards of any materials used or by products produced from the grout or its curing.
3. Large Scale Test Results Showing:
- a. Complete covering of oil surface.
  - b. Bonding to steel.
  - c. Density and variations across length and thickness of pour.
  - d. Growth and leveling of layer as it formed.
  - e. Grout and oil temperature history.
  - f. Shrinkage and/or cracking.
  - g. Other information documenting its ability or inability to satisfy the intent of the system as defined in Section 1.04.
- C. CONTRACTOR shall submit for approval a schedule of the grout pours within 14 days of notice to proceed.

## PART 2 - MATERIALS

2.01 The material components and mix shall be specified by the CONTRACTOR and approved by the GOVERNMENT.

2.02 The grout shall have the following characteristics and requirements:

- A. The grout can be mixed, pumped, and delivered down to the oil surface without altering the characteristics which qualified it for use (refer to Section 1.06).
- B. The grout shall have a specific gravity that is less than 45 lb/ft<sup>3</sup>.
- C. The grout shall not be affected by the crude oil. Resin, filler or aggregate, or any other component of the grout shall not separate from the grout and sink into the crude oil. The crude oil shall not destabilize the mix and cause any material to sink into the crude oil.
- D. Grout shall have a fluidity that allows it to spread across the surface of the crude oil from the injection point(s) and flow around all vertical pipes in the Service Shaft (refer to Figure 1-2), self-level, and completely cover the entire oil surface.
- E. The grout shall harden within 24 hrs of the pour and be ready to accept the next grout (low or high density) pour.
- F. The grout shall develop a minimum shear bond strength to salt and steel of 10 psi 24 hrs after mixing for both clean dry and oil coated interface conditions.
- G. The grout shall develop a minimum tensile strength of 10 psi 24 hrs after the pour.
- H. The grout material shall not shrink more than 1.0 percent in a 24 hr period or develop any cracks.
- I. The maximum exotherm of the grout shall be controlled such that "runaway" or uncontrolled reactions do not occur.
- J. The temperature of the crude oil due to heat released from the epoxy shall not exceed 150°F.
- K. The shelf life of the grout parts or packages shall be at least 2 weeks from date of manufacture.

## 2.03 PREMEASURED AND PREPACKAGED

The epoxy resin, hardener, and blended aggregates shall be packaged and identified with a unique number or mark which identifies the package and batch. Each individual container of material shall be marked with at least the following **information**-material specification number, product designation, name of manufacturer, manufacturer's lot number, date of manufacture, shelf life, and hazard warnings. A material safety data sheet (MSDS) shall be included with each shipment of grout components.

Each lot shall consist of all materials necessary to form a batch or pour of grout. Each batch shall be sampled for Quality Control (refer to Section 3.04). The Quality Control measures will be matched against the minimum properties required in this specification for acceptance.

## PART 3 - EXECUTION

### 3.01 GENERAL

- A. Crude oil storage area and hydrocarbon vapors are isolated from the access level of the mine by existing bulkheads, flanges, valves, and other seals. The CONTRACTOR will maintain isolation as much as is practical as follows:
  - 1. Although the space that will be occupied by the grout plug is isolated from the oil storage cavern and hydrocarbon vapors by the existing grout plug, there is the possibility that hydrocarbon vapors may seep into this space. The CONTRACTOR will therefore regard the grout plug space as potentially containing hydrocarbon vapors. CONTRACTOR will therefore minimize openings between the service level and the grout plug space.
  - 2. The grout fill hole shall be capped and have a seal between the grout fill pipe and the grout fill nozzle and be open only for the minimum time to insert or withdraw the grout fill pipe.
- B. CONTRACTOR shall monitor continuously the quality of air in the raisebores and Service Shaft Drift Areas. The monitoring shall include but not be limited to hydrocarbon vapors and minimum oxygen levels.
- C. The low density epoxy grout plugs in the raisebores shall be completed prior to any pours made down Service Shaft.

### 3.02 CONSTRUCTION

- A. The previously measured, packaged, and identified components will be matched for batch numbers. The matched components

shall be mixed in the mine and pumped down a hose or Tremie pipe on top of the oil to a depth of 12 in. The grout line will be positioned near the center of the shaft or raisebores. The temperature of all grout materials shall be  $80^{\circ}\text{F} \pm 5^{\circ}\text{F}$ .

- B. The end of the injection hose or tremie pipe for placing the grout shall be located not more than 2 ft above the level of the oil, when pouring the first layer of grout. The end of the injection hose or tremie pipe shall be baffled to deflect the grout into a horizontal direction so that it will float on the oil and not plunge into the oil.
- C. Samples from the pour shall be taken for Quality Control. See subsection 3.04 Field Quality Control.
- D. CONTRACTOR shall wait 24 hrs for the grout to set and dissipate the heat of the reaction and then pour another lift of 12 in as described above.
- E. CONTRACTOR will wait 24 hrs after second pour and then perform Plug Permeability Test as described in Attachment 4.02.
- F. If the permeability of plug is verified as acceptable, no additional low density grout pours are required.
- G. Subsequent lifts, if required, shall be poured in 12 in lifts with 24 hr wait periods.
- H. The end of the injection hose or tremie pipe for pouring subsequent layers of grout shall be located not more than 2 ft above the level of the previous layer of grout.
- I. The injection hose or tremie pipe shall be electrically grounded.
- J. During the pour, operations, and wait periods, the fumes from the setting epoxy shall be vented into the existing exhaust ventilation system.
- K. If samples taken from a pour fail to meet the minimum criteria of this specification, CONTRACTOR shall add a 12 in pour, at no cost to the GOVERNMENT for each lift that fails to meet the required minimum specification.

### 3.03 QUALITY CONTROL, ASSURANCE, AND VERIFICATION

- A. The CONTRACTOR shall measure density, exotherm history including time to peak temperature, and spreading factor for each batch of grout before material is placed down hole. The Quality Control tests may be performed on site or at the CONTRACTOR location. The results of the tests must meet or exceed the material requirements to be certified for use. (Refer to Attachment 4.01).

- B. Batches will then be clearly labeled and marked as certified for use and kept in a cool ( $70^{\circ} \pm 20^{\circ}\text{F}$ ), dry environment until used.
- C. Materials must be used no later than two weeks after certifying. After two weeks, the materials shall be retested to recertify the materials for another week. The two week time constraint may be extended if aging studies prove that the material is stable (shelf life) for a longer period of time.
- D. After mixing, but prior to emplacing grout in the mine, a hand specimen will be dropped into a container of Weeks Island crude oil to visually check its ability to float, not adversely react, and spread over the crude oil. This will be done on a batch by batch basis.
- E. CONTRACTOR will test permeability of the grout plug after the second 12 in layer and test each additional layer until the low density plug is verified as adequate according to the criteria and procedures in Attachment 4.02.
- F. The CONTRACTOR shall provide documents showing the beginning and ending elevation of each grout layer.
- G. INSPECTION:
  - 1. Hold Inspection Points:
    - a. Prior to positioning packer.
    - b. Prior to pouring grout.
    - c. Prior to packer release or removal.
  - 2. Witness Inspection Points:
    - a. Positioning of packer in pipe.
    - b. Quality Control tests,
    - c. Pouring of each batch of grout.
    - d. Each stage of the Permeability Test as defined in Attachment 4.02.

#### **PART 4 - ATTACHMENTS**

4.01 Quality Control Tests

4.02 Plug Permeability Test

## Attachment 4.04 - Quality Control Tests

Each batch of materials shall be sampled and tested per instructions below. The Quality Control measures will be matched against the minimum properties required in this specification for acceptance. In addition, to demonstrate that the materials sampled for Quality Control are the same as those used to qualify the grout (Large Scale Test) for use, the Quality Control Test measures must fall within a specified range.

### A. Quality Control Tests

#### 1. Density Determination.

- a. Thoroughly mix components A and B to form 1000 grams of grout. Record start time. Initial temperature of all materials shall be  $80^{\circ}\text{F} \pm 5^{\circ}\text{F}$ .
- b. Preweigh or tare graduated 1000 cc beaker.
- c. Fill beaker to 900 cc level with grout.
- d. Record weight of grout in beaker cc, (W).
- e. Calculate and report density in  $\text{lbs/ft}^3$  as follows:

$$\text{Density (lb/ft}^3\text{)} = (W / 900) \times 62.4$$

#### 2. Maximum Exotherm Temperature.

- a. Position a thermocouple in middle of density sample. Be sure to mark thermocouple wire to assist positioning it in the grout.
- b. Determine the time and temperature at the point of maximum exotherm.
- c. Report maximum exotherm temperature and the time required to reach maximum exotherm temperature. The start time should be the time when the mixing of the grout was started.

#### 3. Reaction with Crude Oil.

- a. Almost fill a 500 cc beaker or other suitable clean container with Weeks Island crude oil.
- b. Measure oil temperature. Before use it must be  $80.0^{\circ}\text{F} \pm 5.0^{\circ}\text{F}$ . Initial temperature of epoxy materials shall be  $80^{\circ}\text{F} \pm 5^{\circ}\text{F}$ .
- c. Preweigh or tare 500 cc beaker with oil and dispense approximately 5.0 grams of grout. Record or calculate the actual grout weight (W1) in grams.

- d. Let grout cure on oil surface for at least 24 hrs. Do not disturb the samples until they have cured.
- e. Remove cured grout samples from the oil surface and inspect edges and surfaces for any chemical or mechanical interactions with the crude oil.
- f. Clean and record weight (W<sub>2</sub>) of sample.
- g. Calculate and report change of sample in percent where:

$$\text{Weight Change (\%)} = [(W_2 - W_1) / W_1] \times 100$$

#### 4. Spreading.

- a. Dispense approximately 20 grams of epoxy grout onto a flat level sheet of release grade Mylar. The initial grout and ambient room temperature must remain at 80.0°F ± 5.0°F until test is completed.
- b. Let grout sample cure on Mylar surface for at least 24 hours. Do not disturb the samples.
- c. Weigh and record sample weight (W).
- d. Measure maximum (L<sub>max</sub>) and minimum (L<sub>min</sub>) dimensions of the sample in inches. Figure A-1 illustrates the dimensions needed.
- e. Calculate and report the spreading factor as follows:
- f. Spreading Factor =  $[(L_{\min} + L_{\max}) / W] \times 18.9$

The spreading factor is a measure of the ability of the grout to laterally spread (avg. length) normalized with respect to weight and is converted to units of ft/lb in the above equation.

#### B. Quality Control Specifications

These specifications must be developed by performing the above Quality Control Tests on the same grout material that Qualified for use (refer to Section 1.06). A low density grout developed and tested at Wil-Cor, Inc. of Pasadena, Texas, has qualified for use by meeting the requirements of this specification. The material is named Armor Plate Ultra Low Density Grout 1000. The specifications of this material are listed below.

1. When tested the density shall be 42 ± 3 lb/ft<sup>3</sup>.
2. When tested the maximum exotherm shall be 198 ± 22°F.
3. When tested the time to maximum exotherm shall be 80 ± 20 minutes.

4. When tested the grout shall harden and float on crude oil with no observable chemical or mechanical reaction with the crude oil.
5. When tested the weight change shall be  $2.0 \pm 3.0$  percent.
6. When tested the spreading factor shall be  $6.9 \pm 2.1$  ft/lb.

If the material tested falls within the acceptable range of values for the property being measured, it may be used down-hole in the raisebores or Service Shaft. Otherwise, it shall not be used and shall be discarded at no cost to the GOVERNMENT.

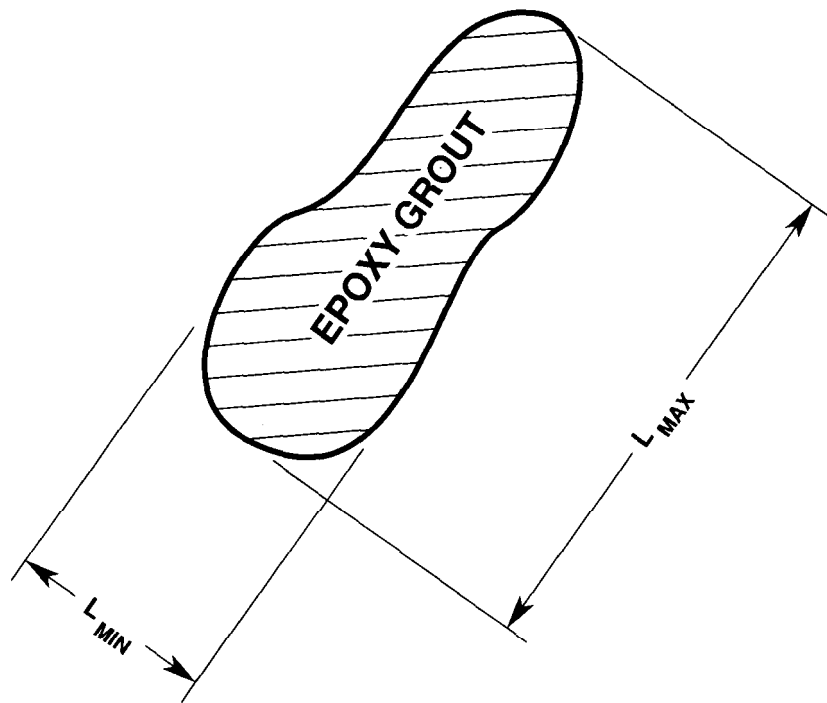


Figure A- 1. Measurements for Spreading Factor. Recommend Use of Vernier Calipers to Measure L **MIN** and L **MAX**.

#### **Attachment 4.02 - Plug Permeability Test**

CONTRACTOR will verify ability of low density epoxy grout to retain high density epoxy grout before it is poured. This test is important to prevent the non-buoyant high density epoxy grout from bypassing the low density epoxy grout plug and sinking into the sump at the bottom of the Service Shaft. The test shall be performed after the second layer of low density grout is placed and cured for 24 hrs, and after each subsequent layer until the permeability of the plug is satisfactory. Test shall be performed in the raisebores and Service Shaft. The three major steps of the test are illustrated in Figure A-2. The procedure is as follows:

1. Allow grout layer to cure, harden, and cool for 24 hrs after pour. Packer will be inflated in a slotted pipe during this time.
2. Using grout pipe or hose, pump a predetermined amount of brine (equivalent to 2 ft deep) on top of the grout. The brine should be pumped onto the plug within 1 hour.
3. Measure and record the depth of the brine using a rod or plumb line. For mechanical measurements a dye may be added to the brine. A low power DC electric circuit well line may also be used provided there is no potential for sparking.
4. If initial measurement shows less than 1 ft of brine, plug is assumed to have leaked and therefore failed this test.
5. Allow brine to rest on plug for 1 hr. Then remeasure the depth of the brine.
6. If more than half of the brine remains as determined from the initial measurement, test is successful. Otherwise, plug failed test\*.
7. Deflate the packer to drain any brine atop the plug. Then reposition (if necessary) and reinflate packer to accept next layer of grout.
8. If test was successful, the next layer of grout can be high density (non-buoyant) grout. Otherwise the next layer shall be low density (buoyant) grout.

\* The criteria for failure is based on the intrinsic permeability of the plug- -a constant independent of fluid type. A typical brine has a viscosity of 2 cps, whereas a dense epoxy grout typically exceeds 100,000 cps. By definition, the hydraulic conductivity of brine is 50,000 times that of dense epoxy. The loss of 1 ft of brine through the plug translates into an expected loss of 1/50,000 ft of epoxy in 1 hr. At later times, the epoxy is expected to cure until hardened.

The criteria for failure may be adjusted provided that the viscosity of the high density grout is measured. Because exothermic heat and

hardening (cross-linkage) are competing influences on viscosity with time, the lowest viscosity may occur sometime after mixing. The viscosity used to set any criteria should be the minimum. This requires multiple time-dependent measurements of the grout after mixing.

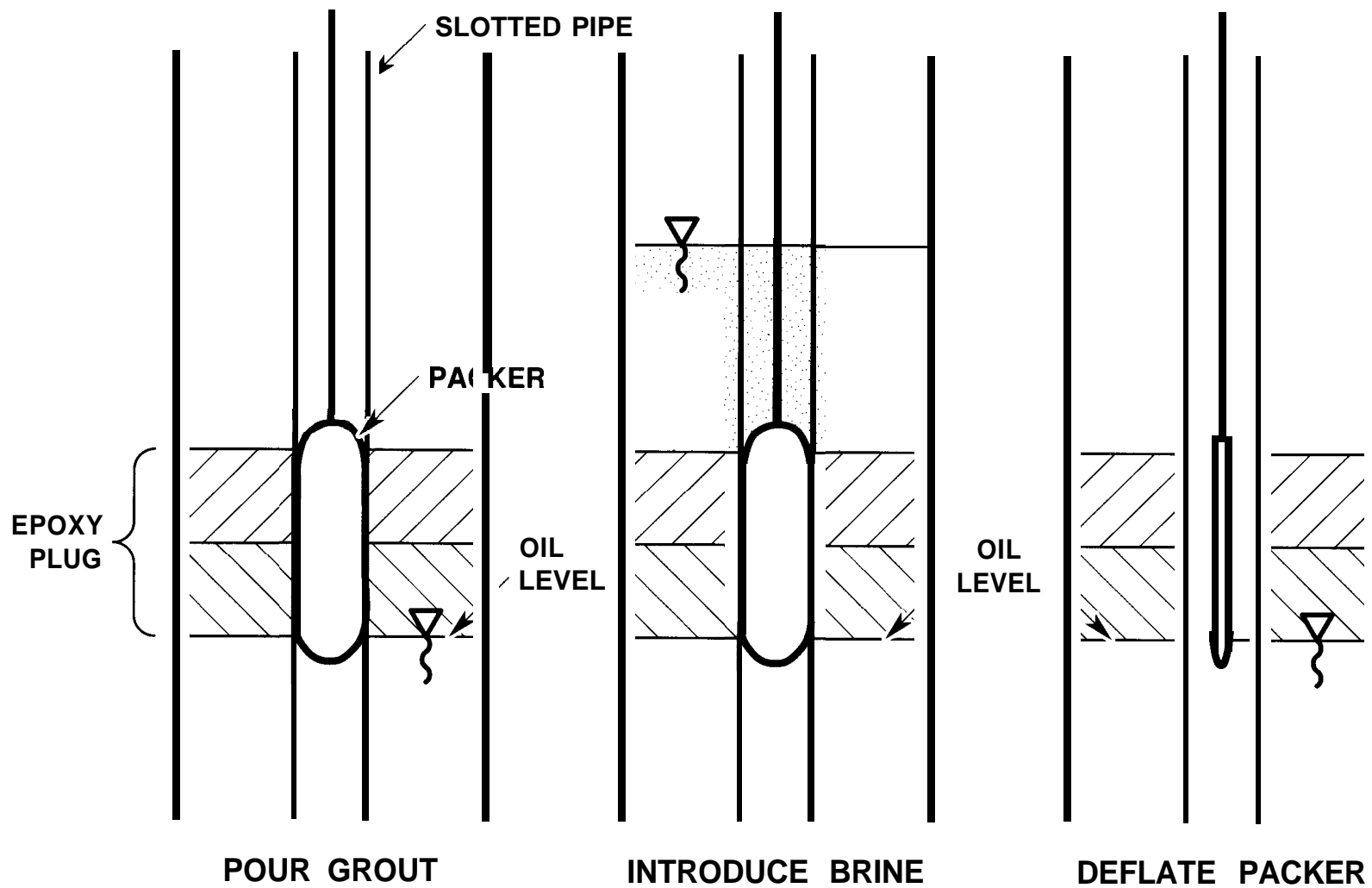


Figure A-2. Permeability Verification Test.

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